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TEXTILE TESTING

Physical, Chemical and Microscopical

by

JOHN H. SKINKLE
Associate Professor of Textile Chemistry
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Second Edition, Revised and Enlarged

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INTRODUCTION TO FIRST EDITION

Textile testing is carried out for one of several reasons and at several stages in the production of the goods. In general, we may say that testing is carried out in order to answer one or more questions: Are the goods satisfactory? Are the goods better or poorer than another lot? What has been the effect of a certain process on the goods? If the goods are unsatisfactory or unusual, what is the cause of the abnormality? It will be seen that to answer all of these questions except the first involves a comparison of two samples; therefore, absolute standards and tolerances are necessary only to answer the first question. The answers to the first two questions are of interest primarily to the consumer; the answers to the last two questions are of interest to the manufacturer.

Where several methods are available the one considered best will be given in detail, and the other methods will be mentioned or considered in less detail. Very often, approximate results may be obtained which are satisfactory for plant use or in a comparison and which require little or no special apparatus. These are especially valuable, since many small plants cannot afford expensive apparatus, and even larger plants occasionally wish to make a test of some sort which is not usually performed because it is unnecessary, and which does not warrant the outlay of any great sum of money.

At the end of each chapter, a bibliography of references is given for each test which is mentioned in the chapter, so that if the reader is interested, he can obtain further information.

INTRODUCTION TO SECOND EDITION

It has been a source of pride to the author that the textile industry has found the first edition of this book valuable enough for general use, and he hopes that this second edition will be received with the same enthusiasm.

The changes which were made were mostly in the direction of adding new material rather than discarding much, and in bringing up to date many techniques and methods. The section on water resistance has been entirely rewritten in view of the experience obtained in World War II. It is hoped that the scheme for the identification of organic finishing agents in Chapter 13 is, a start toward more systematic procedures of analysis in the textile industry. The analysis of fiber mixtures in Chapter 14 as well as the microscopical identifications in Chapter 18, has been revised to include the newer commercial fibers.

Reviews of the first edition show a misconception of the literature references. These are intended for additional information, especially concerning detail, for the research worker; they are not intended simply as the source of the material in the book.

The initial chapter on statistical analysis has been revised to include several more uses for statistics, however, one chapter obviously cannot cover the whole subject, so a compromise has been made which gives the formulae and methods of use empirically and leaves further methods and study to the individual.

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PART I

PHYSICAL TESTING

Chapter 1

STATISTICAL ANALYSIS

Calculation of Test Results

In making physical tests on a textile sample, we must remember that we are dealing with a very variable substance. For example, if we make several determinations of strength on a piece of cloth, we may find that no two tests give the same result. We may average the results, but how accurate is the average? However, formulæ (equations) have been developed, based on the theory of probability, which enable us not only to calculate the result desired but also to obtain other very valuable information. The development of these formulae is too complex to follow, but it is possible to use the formulae with comparatively little trouble. The following symbols will be used in the equations:

n = number of tests made
m = result of any one test
M = arithmetical mean of all the tests
d = deviation of any one test from the mean, regardless of whether it is plus or minus
sd = standard deviation of the tests from the mean
SE = standard error of the mean

The formulae are as follows:

$$M = \frac{\Sigma m}{n}$$

$$d = M - m \text{ or } m - M$$

$$sd = \sqrt{\frac{\Sigma(d^2)}{n}} \text{ (square root of the mean square)}$$

$$SE = \frac{sd}{\sqrt{n - 1}}$$

$$\text{True value} = M \pm SE$$

The standard error should never have more than two significant figures, and the mean should have as many decimal places as the standard error.

The use of the formulae can best be shown by a simple example. Let us suppose that we have made five strength tests on a piece of cloth and the results are:

$$\begin{aligned}
m_1 &= 30.5 \text{ pounds}\\
m_2 &= 31.2 \text{ pounds}\\
m_3 &= 29.3 \text{ pounds}\\
m_4 &= 30.8 \text{ pounds}\\
m_5 &= \underline{32.2} \text{ pounds}\\
\Sigma m &= 154.0 \text{ pounds}\\
n &= 5
\end{aligned}$$

Then,

$$M = \frac{\Sigma m}{n} = \frac{154.0}{5} = 30.8 \text{ pounds}$$

Calculating values of d and d^2

$$\begin{aligned}
d_1 &= 30.8 - 30.5 = 0.3 & d_1^2 &= 0.09\\
d_2 &= 31.2 - 30.8 = 0.4 & d_2^2 &= 0.16\\
d_3 &= 30.8 - 29.3 = 1.5 & d_3^2 &= 2.25\\
d_4 &= 30.8 - 30.8 = 0.0 & d_4^2 &= 0.00\\
d_5 &= 32.2 - 30.8 = 1.4 & d_5^2 &= \underline{1.96}\\
 & & \Sigma d^2 &= 4.46
\end{aligned}$$

Then,

$$sd = \sqrt{\frac{\Sigma d^2}{n}} = \sqrt{\frac{4.46}{5}} = \sqrt{0.892} = 0.946$$

Then,

$$SE = \frac{sd}{\sqrt{n - 1}} = \frac{0.946}{\sqrt{5 - 1}} = 0.47$$

The strength of the cloth then is 30.80 ± 0.47 or, in other words, the chances are very great that the true strength of the cloth is between 30.33 and 31.27.

The formulae may also be used to find out how many tests would be necessary to give a certain standard error.

The standard deviation expressed as percentage of the mean $\frac{(\text{sd} \times 100)}{\text{M}}$ is used to express the variation in the quality of a piece of cloth.

In the example given, the variation would be:

$$\frac{0.946}{30.8} \times 100 = 3.1\%$$

Another use of standard error is to determine whether or not there is a real difference between two samples. The rule is: if the difference between two mean values is equal to, or greater than, twice the larger standard error, there is a real difference between the samples.

Suppose that a sample of the cloth already tested was boiled out in water and the strength determined as 29.90 ± 0.50. Then, 30.80 – 29.90 = 0.90, which is less than twice the larger standard error (0.50). Therefore, there is no difference in the strength, and the treatment cannot be said to have injured the strength.

If, on boiling with another liquid, the strength had become 29.00 ± 0.40, then 30.80 – 29.00 = 1.80, which is more than twice the larger standard error (0.47). In this case, there is a real difference, and the strength has been affected.

The table at the top of page 6 is a complete statistical analysis of an experiment, in which a sample of cotton cloth was tested for strength before and after a certain treatment. It also illustrates the simplest form of calculating and tabulating results.

Conclusion: There is a real difference between the strengths of the two samples. The variation in the strength of the sample after treatment is greater than before treatment. The treatment decreased the strength of the cloth and made it more uneven.

Before			*After*		
m	d	d^2	m	d	d^2
47.0	2.4	5.76	38.6	1.7	2.89
41.1	1.5	2.25	41.2	0.9	0.81
44.4	0.2	0.04	34.6	5.7	32.49
44.8	0.2	0.04	41.4	1.1	1.21
46.2	1.6	2.56	41.2	0.9	0.81
45.8	1.2	1.44	39.8	0.5	0.25
42.2	2.4	5.76	42.0	1.7	2.89
43.5	1.1	1.21	42.7	2.4	5.76
47.4	2.8	7.84	42.8	2.5	6.25
42.0	2.6	6.76	38.6	1.7	2.89
10|446.4		10|33.66	10|402.9		10|56.25
44.6		3.37	40.3		5.63

$$\text{sd} = \sqrt{3.37} = 1.84 \qquad \text{sd} = \sqrt{5.63} = 2.38$$

$$\text{SE} = \frac{1.84}{\sqrt{9}} = 0.61 \qquad \text{SE} = \frac{2.38}{\sqrt{9}} = 0.79$$

$$\text{Value} = 44.64 \pm 0.61 \qquad \text{Value} = 40.29 \pm 0.79$$

$$\text{Variation} = \frac{1.84 \times 100}{44.6} = 4.1\% \qquad \text{Variation} = \frac{2.38 \times 100}{40.3} = 5.9\%$$

$$44.64 - 40.29 = 4.35$$

$$\frac{4.35}{0.79} = 5.5$$

Determination of Validity of a Value

Statistical analysis is also very useful in determining the validity of one value which is markedly different from the others and may be a mistake instead of a valid value. The rule here is to calculate the standard deviation of the set, omitting the doubtful value. If, then, the deviation of the doubtful value is equal to or greater than four times the standard deviation, the doubtful value should be discarded. If, however, the deviation of that value is less than four times the standard deviation, the value should be retained and the standard deviation should be recalculated with the inclusion of the value in question.

Suppose we have the following five determinations, of which the third is questionable:

	m	d	d^2	
	51	1	1	$sd. = \sqrt{\frac{6}{4}} = 1.2$
	53	1	1	
	60—?			
	50	2	4	$d_{60} = 8$
	52	0	0	
M =	52		6	

Since 8 is more than 4 × 1.2, the value 60 should be discarded. If, however, the questionable value had been 56, it should have been retained, since $d_{56} = 4$ which is less than 4 × 1.2.

Kendall Coefficient

Another statistical determination, the Kendall coefficient, is particularly valuable where it is desired to know whether or not a certain type of measurement is correlated with some property which cannot be expressed in figures. Let us suppose, for example, that the *handle* of five samples of cloth is found by a number of experts to be in a certain order. They cannot express the *handle* in figures, but they can say definitely that the handle of one cloth is better than that of another and the handle of the second cloth is better than that of the third, etc. Let us suppose, also, that we have a certain test which we think will measure the *handle* of the cloth and which does give numerical values. First, we arrange the fabrics according to expert judgment. Then, in a parallel column, we arrange the fabrics according to their order by measurement:

Judgment	*Measurement*
1	1
2	3
3	2
4	4
5	5

This shows that the fabric which is first by judgment is also first by measurement, the fabric which comes second by judgment is third by measurement, the one which is third by judgment is second by measurement, and the position of the fourth and fifth samples is the same in both columns. The question now is how closely the measurements are correlated with expert judgment. This is determined by the Kendall coefficient.

We proceed as follows: in a third column, we place a figure which expresses for each fabric the number of fabrics in correct sequence (with regard to this fabric only) which follow. Thus, for the first fabric, all the following are in correct sequence and the figure is 4; for the second fabric, only two are in correct sequence (the 2 should not come after the 3); for the third fabric, there are two correct sequences; for the fourth, there is one; for the fifth, there is none. We add this column to get the correct number of sequences.

Judgment	*Measurement*	*Correct Sequences*
1	1	4
2	3	2
3	2	2
4	4	1
5	5	0
		9

Then,

$$S = 2K - \frac{n(n-1)}{2}$$

where

n = number of samples
K = the correct number of sequences and
S = excess of correct sequences over incorrect ones

$$\text{Kendall coefficient} = \frac{2S}{n(n-1)}$$

In the above case:

$$S = 2 \times 9 - \frac{5 \times 4}{2} = 18 - 10 = 8$$

From this the

$$\text{Kendall coefficient} = \frac{2 \times 8}{5 \times 4} = \frac{16}{20} = 0.80 \text{ or } 80\%$$

This shows that there is an 80% correlation between the value measured and the quality judged by the experts. An exact correspondence would be shown by a value of 1.00 or 100%, and no correlation between the two would be shown by a value of 0.00. A minus value indicates an inverse relationship. The determined value of 80% is considered good enough to warrant adoption of the method as a means of measuring the handle of the cloth. Obviously, the method may also be used for correlating two or more sets of measurements.

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Ezekial, M., *Methods of Correlation Analysis*, John Wiley and Sons, Inc., New York (1930).

Winn, L. J. and Schwarz, E. R., Technical Evaluation of Textile Finishing Treatments—Use of Rank Correlation, *American Dyestuff Reporter*, **29**, 400 (1940).

Chapter 2

MOISTURE DETERMINATIONS

Relative Humidity

Perhaps the most important single factor in the physical testing of textiles is the amount of moisture in the air. Since it affects the amount of moisture in the textile material, it affects nearly every physical property of the goods.

The amount of moisture in the air may be expressed in one of three ways: (1) partial pressure, the actual pressure, in inches of mercury, due to the water vapor alone; (2) absolute humidity, the weight of the water vapor in a unit volume of air, usually in grains per cubic foot; (3) relative humidity, the per cent saturation of the air either one of the previous values compared to the maximum possible value of humidity at the temperature in question. It is in relative humidity that we are principally interested, since it seems to be the relative humidity rather than the actual moisture which affects the moisture content of the textile sample.

Relative humidity may be defined by either of two equations:

$$RH = 100 \frac{e}{E}$$

or

$$RH = 100 \frac{h}{H},$$

where

RH = relative humidity, per cent
e = actual vapor pressure in inches of mercury
E = maximum vapor pressure in inches of mercury
h = actual absolute humidity in grains per cubic foot
H = maximum absolute humidity in grains per cubic foot

The values of E and H may be obtained from Table I either directly or by interpolation, but the values of e or h must be determined experimentally.

TABLE I
MAXIMUM VAPOR PRESSURE AND ABSOLUTE HUMIDITIES

Temperature °F.	*Vapor Pressure Inches of Mercury*	*Absolute Humidity Grains per Cubic Foot*
32	0.18	2.12
35	0.20	2.38
40	0.25	2.86
45	0.30	3.44
50	0.36	4.11
55	0.44	4.89
60	0.52	5.80
65	0.62	6.85
70	0.74	8.07
75	0.88	9.46
80	1.03	11.06
85	1.22	12.88
90	1.42	14.95
95	1.66	17.30
100	1.94	19.97
105	2.25	22.97
110	2.60	26.34
120	3.45	34.37
130	4.53	44.36
140	5.89	56.69
150	7.58	71.74
160	9.66	89.99
170	12.21	111.9
180	15.30	138.0
190	19.02	169.0
200	23.47	205.3
210	28.75	247.8
212	29.92	257.1

DETERMINATION OF RELATIVE HUMIDITY

The following methods may be used to determine relative humidity:

1. Chemical method
2. Dew-point method
3. Hair-hygrometer method
4. Wet and dry bulb hygrometer method
   a. With stationary-type hygrometer
   b. With sling psychrometer

Chemical Method

The chemical method involves passing a measured volume of air through a drying agent such as phosphorus pentoxide or calcium chloride, the drying bulb being weighed before and after the experiment. The increase in weight, converted to grains and divided by the number of cubic feet of air passed through gives the actual absolute humidity (h), from which:

$$RH = 100 \frac{h}{H}$$

where H is determined from Table I. This method is the most accurate of any, even more accurate than is necessary for textile purposes however, it involves the use of an analytical balance for weighing the moisture and an accurate meter for measuring the air, both of which are expensive and delicate, requiring a specially trained man to operate. Chemical methods, therefore, are seldom used in textile testing.

Dew-Point Method

The dew-point method is the next most accurate method. It consists in cooling a polished metal container of water slowly until moisture just condenses on its outer surface. This occurs at a temperature which is called the dew point. The dew point is, then, the temperature at which the air would be saturated if cooled without loss of water vapor. An example will show best how relative humidity is calculated from dew point. Using Table I, let us assume that the temperature of the air in a room is 70° F. and its dew point, determined as above, is 50° F. The air would be saturated at 50° and would, therefore, have an absolute humidity of 4.11 grains per cubic foot (h); at 70° F., the maximum possible absolute humidity is 8.07 grains per cubic foot (H). Then

$$\mathrm{RH} = 100\,\frac{\mathrm{h}}{\mathrm{H}} = 100 \times \frac{4.11}{8.07} = 51\%.$$

As stated before, the dew-point method is next in accuracy to the chemical method but, since it requires considerable care and the use of a cooling medium, it is not very convenient for use in a plant.

Hair-Hygrometer Method

Hair hygrometers are very frequently met with now, in the form of small table-top or wall hygrometers. They operate by the change in length of a hair or band of hair acting through a link motion on a pointer. The stretch of a hair with change in relative humidity is approximately a straight line only over the middle portion of the scale, the response of the hair to changes in relative humidity is very slow and the link motion always has some play in it. For these reasons, the hair hygrometer is the least accurate instrument for determining relative humidity and it requires frequent calibration. It is useful only for rough indications of rather large changes in relative humidity and should not be used for laboratory work.

Wet and Dry Bulb Hygrometer Method

The wet and dry bulb hygrometer operates on the principle that if a thermometer is surrounded by a film of water and the air around it is not saturated, water will evaporate from the film at a rate proportional to the difference between saturation and the actual relative humidity. This evaporation requires heat which is taken from the bulb of the thermometer, so that the wet-bulb thermometer will read less than the dry-bulb thermometer by an amount inversely proportional to the relative humidity. It should be noted that the wet-bulb reading is not the same as the dew point.

The wet and dry bulb bulb hygrometer is convenient and easy to manipulate. It is accurate to 1% relative humidity (fractions of a per cent are not given) and so is the most common type of hygrometer used. There are two main types: the stationary and the sling psychrometer.

Stationary Type

The stationary type has the thermometers fixed in position. The wet bulb is moistened by a wick which dips into a container holding water, preferably distilled water. Since the air surrounding the wet bulb tends to be of higher relative humidity than the air in the rest of the room, it is desirable to fan hygrometers of this type before reading the thermometers. The Hygrodeik is the most convenient instrument of this type, since a diagram and pointer for calculating the relative humidity are incorporated in the apparatus. Other forms of apparatus have a fan in the instrument for drawing air past the thermometers. These are desirable if so arranged that the heat of the motor does not affect the thermometers.

Sling Psychrometer Method

This is an instrument which is free from the objectionable stationary air film and yet uses the simple wet and dry bulb principle. It consists of a pair of matched thermometers fastened to a rigid frame which can be whirled through the air. One of the thermometers is covered with a fine wick of silk, rayon, or mercerized cotton, which is wetted with distilled water before use. This instrument gives quick and accurate readings of the relative humidity but is not continuously recording.

In wetting the wick, distilled water at a temperature just above the wet-bulb temperature (room temperature is satisfactory) should be used for reasons illustrated in Fig. 1. If water colder than the wet bulb is used, the temperature rises with only a point of inflection at the wet-bulb temperature. If too warm water is used, all of it is evaporated and the temperature starts to rise again before wet-bulb temperature is reached. If water only a few degrees warmer than the wet bulb is used, however, the temperature drops rapidly to a long constant-reading minimum before rising again.

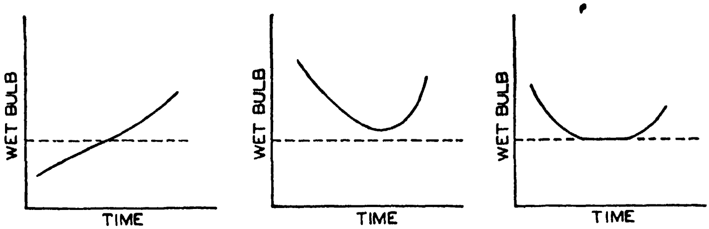

FIG. 1. Effect of Temperature of Water on Wet-bulb Reading

a. Water is below wet-bulb temperature

b. Water is much above wet-bulb temperature

c. Water is slightly above wet-bulb temperature

The thermometers should be capable of being read to $\frac{1}{4}$° F. if the full accuracy of the instrument (the nearest 1% relative humidity) is to be realized; for plant use, however, thermom-

TABLE II

RELATIVE HUMIDITIES BY SLING PSYCHROMETER

Wet-bulb Depression	*Dry-bulb Temperatures* °F.								
	60	*65*	*70*	*75*	*80*	*85*	*90*	*95*	*100*
2	89	90	90	91	91	92	92	93	94
4	78	80	81	82	83	84	85	85	86
6	68	70	72	74	75	77	78	79	80
8	58	61	64	66	68	69	71	72	73
10	48	52	55	58	61	63	65	66	67
12	39	44	48	51	54	57	58	60	62
14	30	35	40	44	47	50	52	54	56
16	21	27	33	37	41	44	47	49	51
18	13	20	25	30	35	38	41	43	46
20		12	19	24	29	33	36	38	41
22			12	18	23	27	31	34	37
24				12	18	22	26	29	32

eters accurate to ½° F. are satisfactory. The sling should be whirled at such a rate that the wet bulb has a linear velocity of 10 to 20 feet per second. After the wet bulb is moistened with distilled water at room temperature, the instrument is whirled for 30 seconds and the thermometers are read (wet bulb first, instrument held away from body). The instrument is then whirled for 20 seconds and read again; this is repeated until constant readings are obtained.

The relative humidity may then be obtained roughly by the use of Table II or more exactly by the use of the data of Table I and the following formula:

$$e = e' - 0.000367P(t - t')\left(1 + \frac{(t' - 32)}{1571}\right)$$

where, in addition to the symbols already used,

t = dry-bulb temperature in °F.
t' = wet-bulb temperature in °F.
P = barometric pressure in inches of mercury
e' = maximum vapor pressure at t (Table I)

As an example, suppose that the barometer is 30.0 inches of mercury, dry-bulb temperature is 80° F. and wet-bulb temperature is 70° F. Then,

$$\begin{aligned} e &= 0.74 - .000367 \times 30.0(80 - 70)\left(1 + \frac{70 - 32}{1571}\right) \\ &= 0.74 - .000367 \times 30.0 \times 10 \times 1.024 \\ &= 0.74 - .11 = 0.63 \end{aligned}$$

$$RH = 100\frac{e}{E} = 100 \times \frac{0.63}{1.03} = 61\%$$

From Table II, also, the value of the relative humidity is 61%.

CONTROL OF CONDITIONS FOR TESTING

Since temperature and relative humidity have such important effects on the physical properties of textiles, it is necessary that the conditions at which samples are tested are rigidly controlled. The effect of temperature is so small compared to the effect of relative humidity that the control of temperature may be unnecessary, although it is often desirable if the volume of work done warrants it. Many types of thermostats are available, and one should be selected which is applicable to the system of heating or cooling used. If the temperature does not vary more than 10° F., no regulation is necessary. If the variation is more than this, it may be compensated for by the following rules:

To maintain a constant amount of moisture in cotton goods, increase the relative humidity 1% for every 10° F. above standard temperature.

To maintain a constant amount of moisture in woolen goods, increase the relative humidity 2% for every 10° F. above standard temperature.

The usually accepted standard conditions are 70° F. ± 2° and 65% ± 2% relative humidity. The humidity is set at this comparatively high value because it is much easier to humidify air than to dehumidify it.

Crude Method

The crudest way of obtaining a given humidity is to wet the floor and walls of a small room or set out open pans of water. This gives very little control and tests must be made very quickly while the air is at the desired humidity. Since, as we shall see later, most textile material requires several hours to come to equilibrium with the atmosphere, this method is very unsatisfactory.

Use of Acid and Salt Solutions

A method of obtaining constant relative humidity in a comparatively small space is to make use of the vapor pressure of various salts or saturated solutions of salts. More convenient still is the use of solutions of acids or salts which can be analyzed easily for composition, such as solutions of sulfuric acid, phosphoric acid, or calcium chloride. The writer has used solutions of sulfuric acid for this purpose, since the concentration of the acid can be easily determined by a simple titration. Table III gives the relative humidities corresponding to various concentrations of sulfuric acid.

TABLE III
RELATIVE HUMIDITIES ABOVE SULFURIC ACID SOLUTIONS

Relative Humidity %	*Concentration of Sulfuric Acid* % *Weight/Volume*	*Relative Humidity* %	*Concentration of Sulfuric Acid* % *Weight/Volume*
5	69.0	55	41.5
10	64.0	60	38.6
15	61.0	65	35.9
20	57.7	70	33.7
25	55.2	75	30.8
30	52.5	80	27.1
35	50.3	85	22.5
40	48.0	90	16.4
45	46.0	95	9.8
50	43.9	100	0.0

Use of Hygrostats

Best of all, if the amount of testing done warrants the expense, is the use of hygrostats or psychrostats which control the wet- and dry-bulb temperatures automatically, by adding moisture to the air in quantities just sufficient to keep the room at the desired humidity. The moisture is added to the air by means of sprays in the room at the ceiling, or the air is drawn from the room through a compartment or cabinet where it is humidified by sprays and blown out into the room again. The last method is preferable because it gives a circulation of air which tends to eliminate dry or over-moist pockets.

The controlling element of the psychrostat or hygrostat may be one of two types. An older type, which is still in use, depends on a filament of hair, viscose, or gut, which lengthens with increasing humidity and so regulates a compressed air valve which, in turn, controls the sprays. This type of control is open to the same criticisms as hair hygrometers, i.e., slow response to changes in humidity and the necessity of frequent recalibration. A more recent type of control depends upon the use of two thermometer bellows, an atomizer, spraying over one, (wet bulb) draws air over the other (dry bulb). The dry bulb is connected by a link motion to the seat of a slide valve, and the wet bulb to the slide valve itself. The two act together to regulate the flow of compressed air which, in turn, operates the sprays of the humidifier.

Regain

In expressing the amount of moisture present in a textile material, two methods have been used: per cent moisture and per cent regain. The per cent moisture is calculated in the same way as any expression of percentage, that is, the weight of the component divided by the total weight of the sample and multiplied by 100. The per cent regain has been defined as the weight of moisture picked up (or regained) by 100 parts of dry material. This definition is misleading, however, in that this process is never carried out in the direction stated but always in the reverse direction. We shall define regain, therefore, as the weight of moisture in a sample divided by the dry weight of the sample and multiplied by 100. Per cent moisture is used mostly by chemists; per cent regain is used mostly by textile technicians.

To express the results in formula form, let

a = weight of sample with moisture
b = weight of sample after drying
M = per cent moisture
R = regain

Then,

$$M = \frac{100(a - b)}{a} \qquad R = \frac{100(a - b)}{b}$$

The interrelationships of M and R are shown by the following:

$$R = \frac{100M}{100 - M} \qquad M = \frac{100R}{100 + R}$$

Obviously, if we know either M or R, we may find the other. Also, of the three quantities a, b, and R, if we know any two, we may find the third:

$$a = b\left(1 + \frac{R}{100}\right) = b\left(\frac{100 + R}{100}\right)$$

$$b = \frac{a}{1 + \frac{R}{100}} = a\left(\frac{100}{100 + R}\right)$$

METHODS OF DETERMINATION

There are five principal methods which have been used for the determination of regain:

1. Xylol method
2. Relative humidity in equilibrium with sample
3. Vacuum desiccator with drying agent
4. Volumetric titration method
5. Drying ovens
    a. Low temperature, using dry air
    b. High temperature, using air from room

The first four of these methods are comparatively unimportant.

Xylol Method

The xylol method depends upon distilling from a flask containing the sample and a large volume of xylol. The moisture distills over with the xylol and may be collected in a measuring tube or graduate. This method requires a large sample, bulky apparatus, and recovery of the xylol used.

Relative Humidity in Equilibrium with Sample

The determination of relative humidity in equilibrium with a sample as a measure of the regain of the sample has been used for quick but rather inaccurate determinations. One method is to place the sample in a sealed vessel with a hygrometer attached. Another method is to place the sample in a closed circuit through which air is circulated, a hygrometer indicating the relative humidity.

Vaccum Desiccator

The third method mentioned was used by Urquhart in making the most accurate regain determinations to date. The sample was placed in a desiccator containing phosphorus pentoxide and the desiccator was then evacuated. At the end of a week, the moisture had been completely removed from the sample. This is the most accurate method, but the time required makes its use impractical for industrial purposes.

Volumetric Titration Method

The titration method involves treatment of the sample with synthetic methanol and then titration with Fischer reagent (a solution of iodine, sulfur dioxide, and pyridine in methanol). This method is quick and suitable for use when many determinations are being made each day. However, it has the disadvantage that the reagent is not stable and requires standardizing each day. This method is applicable to cotton but probably not to wool.

Oven Method

The oven method is the best method to use, considering the factors of accuracy and speed of determination. When using the oven method at temperatures of 220–230° F. (105–110° C.), air from the room may be used to dry the samples. This is preferable, in most cases, to the low-temperature method of using dried air at 140° F. (60° C.), because it avoids the use of air-drying apparatus which must be renewed constantly. It is quicker and as accurate as the low-temperature method when only the textile material is present with or without the usual additional substances. In a few cases, where certain substances are present on the material, such as some more volatile oils or substances which might react with the fiber at high temperatures, the low-temperature method is desirable. On the whole, however, the high-temperature oven may be considered as the standard method of obtaining regain.

Using the high-temperature oven, the time required to reach dryness varies with the type of fiber and weight of fabric. All fabrics have a drying curve similar in shape to Fig. 2 which shows that most of the moisture is lost very rapidly but that the last portions are held rather tenaciously by the fiber. In all ordinary cases, drying is complete in 1 hour, but 1½ hours should be allowed for safety. Better still, fabrics should be dried to constant weight, that is, until two weighings, 10–15 minutes apart, show no further loss in weight. In the case of some heavy fabrics or felts, this may be a very long time. If we compare regains obtained in this way with regains obtained by the more accurate but very slow vacuum-desiccator method we find that,

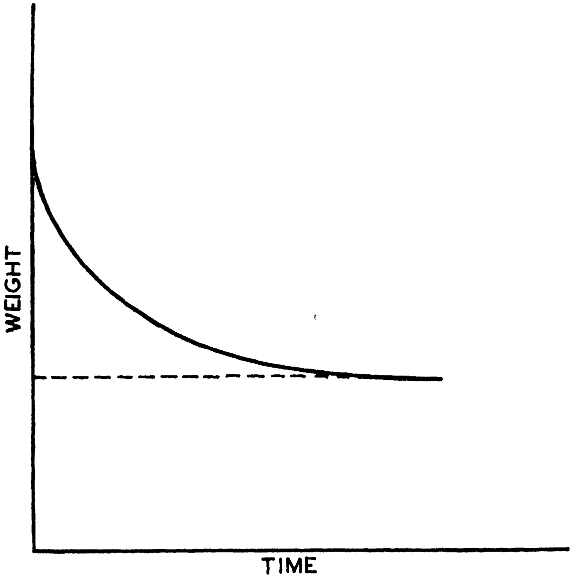

FIG. 2. Drying Curve

in the oven, wool does not lose all of its moisture, whereas cotton loses a little more weight than there is moisture present. This excess loss is not due to decomposition of the cotton, however, but is due to volatilization of some of the impurities present, as shown by the fact that in cotton, which has been boiled out or bleached thoroughly, this loss of weight is insignificant. In view of the fact that the true regains are not given exactly by the oven method, although the error is very small, there is a growing tendency to refer to samples dried in the oven as *oven-dry* rather than as *bone-dry*, which is the old term.

There are two general procedures used in determining the dry weight of a sample:

1. Dry the sample in the oven; transfer it to a weighing bottle and weigh it on a balance outside of the oven.
2. Dry the sample in the oven and weigh it while it is still in the oven.

The method used depends upon what kind of apparatus is available. The ordinary chemical drying oven is found in nearly all chemical laboratories and is very cheap and easy to use. The analytical balance is also found in any well-equipped chemical laboratory. The weighing bottle (a glass bottle with a ground-glass stopper) is dried, cooled in a desiccator, and weighed. This weight is a constant and may be recorded in the laboratory log-book opposite the number of the weighing bottle. The sample is placed in the weighing bottle, and the whole is placed in the oven with the cover of the weighing bottle off (usually tipped over at right angles). After the sample is dried the required length of time, the cover of the weighing bottle is placed in position. The weighing bottle and its contents are cooled in a desiccator and then weighed on a balance. The weight of the bottle is then subtracted from the total weight of the bottle and its contents, and in this way, the dry weight of the sample is obtained.

This method has the disadvantages that it does not readily lend itself to progressive weighings to determine when constant

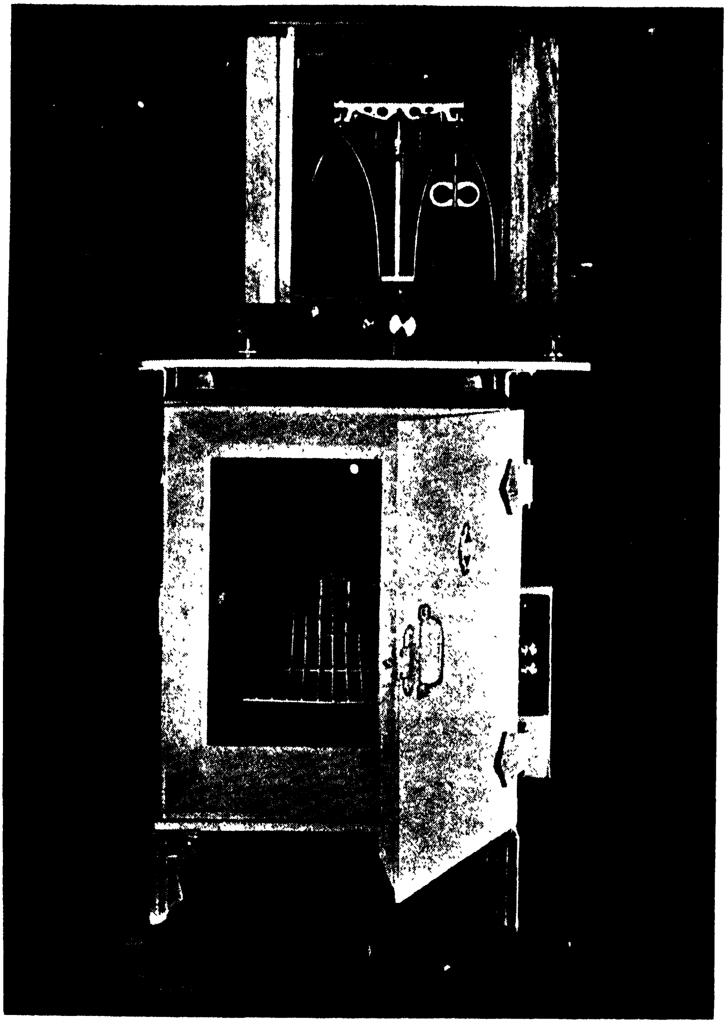

FIG. 3. Oven and Analytical Balance Combination

weight is reached and that there is a possibility of picking up moisture during the weighing. However, it has the advantage of requiring apparatus which is ordinarily present in a laboratory.

A better procedure, when the apparatus is available, is to weigh the samples while they are still in the oven. The apparatus ordinarily sold and used for this purpose is a combination of an oven and a balance which is accurate to 1 centigram or to $\frac{1}{8}$ grain. However, where chemical testing is also to be done, or where it will be necessary to work with small weights of sample, or where small differences in weight will be measured, it is much better to have a combination oven and analytical balance (Fig. 3).

In this type of apparatus, the balance is mounted on top of the oven. A rod attached to the left-hand pan of the balance passes down through an opening in the top of the oven and ends in a hook on which may be hung one of a number of baskets of the same weight. A counterpoise equal in weight to the rod and basket is placed on the right-hand pan of the balance. The sample is placed in a basket, dried in the oven, and weighed on the balance at the end of an hour. The weighing is then repeated at intervals of 10 or 15 minutes until constant weight is reached. This is the oven-dry weight. If an analytical balance is used, one of the chainomatic type is very desirable for quicker weighing because of the absence of small weights.

There are also small sample driers on the market, which dry with a blast of hot air in a few minutes. These give very quick results and are suitable where approximate values of the regain are satisfactory, as in many of the chemical determinations. Preferably, they should be of such form that the sample is dried in a container which can then be closed immediately with an air-tight cover for weighing.

FACTORS AFFECTING REGAIN

Relative Humidity

The effect of relative humidity upon regain may be shown best by a graph (Fig. 4). This shows that for any one material, starting with dry air, the addition of only a small amount of moisture to the air results in considerable increase in regain. Then follows a period, indicated by an almost straight line, in which the regain is proportional to the relative humidity. Finally, when the air is nearly saturated, further additions of moisture to the air result in large increases in regain.

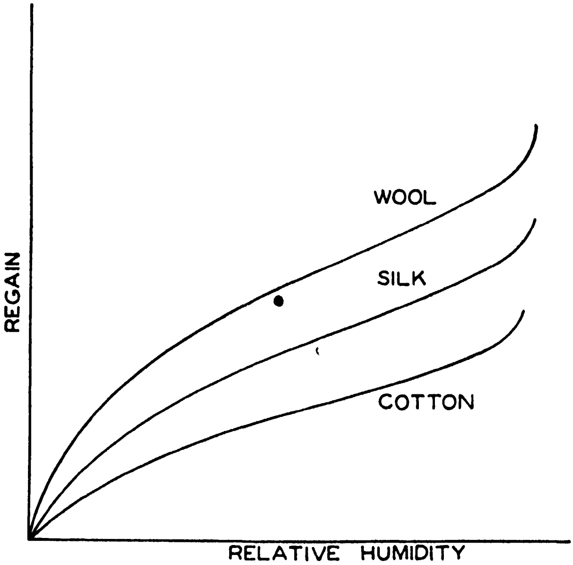

FIG. 4. Effect of Relative Humidity on Regain

A colloid chemist would immediately recognize the shape of this curve as typical of the process of adsorption of a vapor by rigid porous gels, such as silica gel and stannic oxide gel, or charcoal. Since the moisture is being adsorbed from a mixture of air and water vapor, a colloid chemist would also expect the phenomenon of hysteresis, and accurate determinations have shown that it occurs (Fig. 5). Hysteresis is a general term referring to the phenomenon of obtaining different results in a process, according to the direction from which equilibrium is approached. In this particular case, hysteresis manifests itself as two different values of regain for a material in contact with air of a certain relative humidity depending on whether we start with the fabric drier or moister than equilibrium.

If we are testing a sample of wool at standard conditions (70° F., 65% RH) and start with dry wool, we will find that the wool will not pick up more than 14.5% regain. The same sample of wool, if moistened and allowed to come to equilibrium, will not have less than 16.5% regain. This difference in regain would cause considerable difference in the physical properties, so that it is not enough to say what standard conditions are. We must also say how we reach standard conditions. The generally accepted method is the one specified by the A.S.T.M., i.e., "moisture equilibrium shall be approached from the dry side."

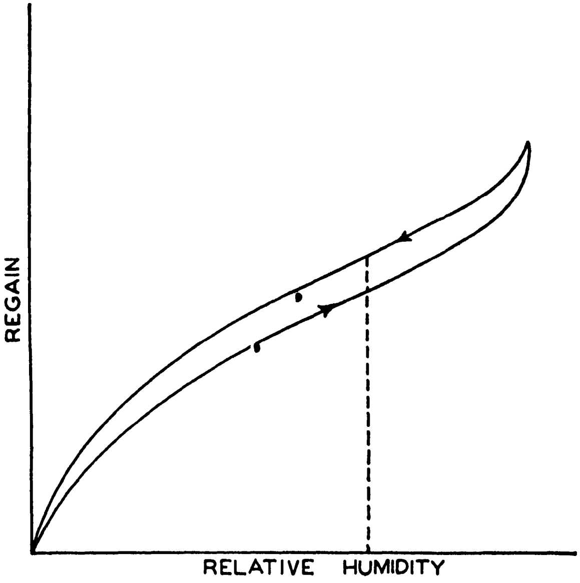

FIG. 5. Hysteresis Effect

Temperature

Regain is comparatively unchanged at room temperature. As indicated in the discussion of relative humidity, temperature changes of 10° F., or more, may be compensated for by a small change in relative humidity; temperature changes of less than 10° F. are negligible.

Time

Textile materials exposed to standard conditions of temperature and relative humidity will require varying periods of time to come to equilibrium. If the material is loose fiber, loosely twisted yarn, or a light porous fabric, 2 hours are sufficient; heavy duck may require up to 6 hours. Samples of hard felt have taken weeks and even months. In order to be certain that equilibrium is reached, it is wise to weigh a small piece of material at intervals until no further increase in weight occurs, after which the material is in equilibrium.

Mechanical Form and Treatment

The previous treatment of the sample and its mechanical form also affect the regain. Cotton taken from the interior of a bale, or wool from the interior of a hard felt, has considerably less moisture than samples not under pressure. Cleansing treatments also alter the regain. For example, at 60% relative humidity and 25° C., raw wool had a regain of 10.9%, the same wool, after washing, had a regain of 13.7% and after combing, the regain was 13.0%; a raw silk had a regain of 7.7%, after degumming, the regain was 10.5%; a sample of cotton had a regain of 7.3%, after bleaching, it had 6.3% regain. Conversely, oiling wool, which is to be combed, decreases the amount of moisture which the wool can hold at any given set of humidity conditions. Whether the material is in the form of loose fiber, yarn, or cloth also affects the regain. For example, at standard conditions, loose cotton will hold 8.5% regain, cotton yarn 7.0 to 7.5%, and cotton cloth about 6.5% regain.

Table IV gives the standard regains for different fibers in various mechanical forms. These standard regains are approximately the regains of the fibers at standard conditions of 70° F. and 65% relative humidity. The values are those which have been officially adopted where this is possible. In a few cases, the values which are generally accepted but which are not official are given.

TABLE IV

STANDARD REGAINS OF TEXTILE MATERIALS

Cotton fiber	8.5
Cotton yarn	7.5
Cotton cloth	6.5
Cotton tire cord	6.5
Mercerized cotton yarn	8.5
Nylon	4.5
Acetate rayon	6.5
Viscose, cupra, rayons	11.0
Silk	11.0
Casein	13.0
Mohair	10.0
Camel hair	13.0
Wool fiber (scoured)	13.6
Woolen yarn	13.0
Worsted (oil spun, Bradford)	13.0
Worsted (dry spun, French)	15.0

CONTROLLING REGAIN EFFECTS

Since the regain is affected by so many variables, it is obvious that its effect must be eliminated in some way during the testing. This has been done in the following manners:

1. Testing oven-dry
2. Testing wet
3. Testing at standard conditions (or standard regain)
4. Testing at room conditions, measuring the regain, and correcting to standard regain or to oven-dry condition

Testing Oven Dry

Testing oven-dry is impracticable for such determinations as strength or twist because of the instant rapid adsorption of moisture that occurs as soon as a sample is exposed to the air. Weighings, however, are most accurate when made oven-dry. All chemical tests are based on oven-dry weighings and such determinations as counts of yarn or weight per unit area of cloth are most accurately made if the weighings are made when the sample is oven-dry.

Testing Wet

Testing in the wet condition, especially strength and stretch testing, was very common in the early days of textile test when the effect of relative humidity was known but nothing had been done to allow for it. The results obtained, however, were not always comparable with the results obtained under normal conditions, so that the practice is obsolete now, except for the case of rayons which are tested for strength both wet and at standard conditions.

Testing at Standard Conditions

Testing at standard conditions or at standard regain is by all means the most accurate and convenient, except for weighings which are better done in the oven-dry condition. Expensive apparatus for the control of humidity is necessary, however, and small plants or laboratories, where textile testing is not done on a large scale, may get along very well by testing at whatever conditions prevail and correcting to standard conditions.

Testing at Room Conditions

In many chemical processes, especially, the effect of a certain treatment is shown by testing a sample before and after the process; since a comparison is all that is necessary, standard conditions are not necessary. Testing can also be carried out at conditions other than standard by testing a standard sample at the same time and referring all other tests to this sample.

In chemical testing, very often, two samples are weighed out at once; on one sample the test is carried out and on the other sample a moisture determination is made. The test sample is then corrected for the regain found in the second sample. This is done, for example, in the case of copper number determinations because heating a sample to get the dry weight might cause more oxycellulose or hydrocellulose formation than is really present at room conditions.

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Chapter 3

FIBER TESTING

Staple and Grade

Staple is the length of a textile fiber but, as commonly used, it means the average length of a sample of fiber. Staple is usually associated with the grade of the fiber, because the same person usually determines both qualities. There is, however, a distinction between staple and grade which is important in testing because one is measurable and the other is the expression of opinion of an expert and is not measurable except by an expert. *Grade*, as used to describe a textile fiber, is a summation, in the mind of the person examining the material, of the color, handle, evenness, staining and fineness of the fiber. The ability to arrive at this summation can be acquired only by long experience and comparison with standards. However, this method of testing seems to be quite reliable because expert graders, working independently, will nearly always arrive at the same result.

Table V gives the standard grades for cotton, wool, and silk. Cotton has five grades ranging from Middling Fair, which is the best grade, to Good Ordinary, which is the poorest grade. Wool has seven grades ranging from Fine (the best) to Braid (the poorest) in the American system. In England, and to an increasing extent in the United States, the practice of grading recognizes a greater number of divisions, originally based on the finest yarn to be spun from the fiber, but now more or less formalized. The fineness of the fiber is the most important factor in grading wool. A microscopic projection method of measuring this fineness has been devised and is in more or less general use. The grading of silk varies with the locality from which the silk comes. The system in use on Japanese silk is given in Table V and consists of seven grades ranging from Crack Double Extra, which is the best, to No. 1 to 1½, which is the poorest. The grading of fibers should not be attempted by the ordinary tester but should be left to an expert. The tables are given so that the meaning of the reported terms may be understood.

TABLE V
STANDARD GRADES OF FIBERS

Standard Grades of Cotton	*Standard Grades of Japanese Raw Silk*
Middling Fair	Crack Double Extra
Good Middling	Double Extra
Middling	Extra
Low Middling	Best No. 1 to Extra
Good Ordinary	Best No. 1
	No. 1
	No. 1 to 1½

Standard Grades of Wool and Wool Top

American Grades	English Grades
Fine	80's, 70's, 64's
½ Blood	60's, 58's
⅜ Blood	56's
¼ Blood	56's, 48's
Low ¼	46's
Common	44's
Braid	40's, 36's

STAPLING

Stapling, on the other hand, is an art which may be fairly easily learned. The following methods are in general use:

1. Hand stapling
   a. Cotton
   b. Wool
2. Machine stapling
   a. Ball
   b. Baer
   c. Suter-Webb

Hand Stapling

Hand stapling of cotton can be readily learned by observation, or from the following directions as given by the U. S. Department of Agriculture:

1. Grasp in the two hands a tuft of cotton of a size convenient for the purpose (about $\frac{1}{4}$ of an ounce), holding it firmly between the thumb and forefinger of each hand, with the thumbs placed together, the fingers being turned in towards the palms of the hands, and the middle joints of the second, third, and fourth fingers of each hand touching the corresponding joints of the fingers of the other hand, so as to give a good leverage for *breaking* the cotton.

2. Pull the cotton slowly apart with about the same leverage of each hand on the joints of the fingers, separating the tuft of cotton into two parts. Discard the part remaining in the right hand.

3. Grasp with the thumb and forefinger of the right hand, the end of the tuft of the cotton retained in the left. The point of pressure on the cotton in the left hand is just below the joint of the thumb and at the nail joint of the forefinger. With the right hand draw a layer of fibers from the cotton held in the left hand. Retain in the right hand the layer so drawn.

4. Repeat this operation four or five times, placing each successive layer directly over the fibers previously drawn, using care to see that the ends of all the layers are even with each other between the thumb and forefinger of the right hand.

5. After discarding the cotton in the left hand, hold the fibers thus obtained between the thumb and forefinger of the right hand and smooth them with the thumb and forefinger of the left hand. Place these fibers on a flat horizontal surface with a black background.

6. Block off the ends of the fibers with a cotton stapling rule so as to indicate the length of the bulk, or body, of the fibers. Then measure the distance between the blocked-off ends.

The result is, of course, the average staple of the cotton and does not give any idea as to the number of fibers of each length.

In the stapling of wool, it is necessary to get some idea of the distribution of fiber lengths as well as the average staple. In the case of wool, also, we have longer fibers which are easier to handle singly or in small numbers. Wool stapling, therefore, consists of laying out all the fibers from a sample on a proper background in the order of their lengths. The steps in stapling a sample of top are as follows:

1. On a piece of black velvet, mounted rigidly on a board, draw a chalk line for a base.
2. Square up the end of the top by pulling out the loose fibers.
3. Grip the square end of the top between the thumb and forefinger of the left hand and pull out a tuft of fibers free from the rest of the top, being careful not to break the fibers. This new end will be long and tapering.
4. Transfer the square end to the right hand and, very slightly, twist together the longest fibers in the tapering end.
5. Place the top of the longest fibers on the chalk line, hold with one finger of the left hand and slowly pull the main tuft away with the right hand, at right angles to the base line. The fibers should cling to the black velvet.
6. Repeat with the next longest fibers, alongside of the first set, and continue until all the fibers are on the velvet. Try to get a uniform density of fibers throughout.

For ordinary mill routine, the average length of staple is determined by placing a rule parallel to the base line and in such a position that there is as much long fiber above as short fiber below. This average length is read, also the longest length. For a somewhat more accurate determination of average staple, a tracing of the fiber layout may be made. The area, measured with a planimeter, divided by the length of the base would then be the average staple. A still more accurate method would be to take all fibers between certain length measurements and weigh them, repeating this operation for the same length interval all along the array. The average length of each group would then be multiplied by the weight of the group. These products added up and divided by the total weight of the sample would give the average staple.

Machine Stapling

The wool-stapling method gives much more information than the cotton-stapling method, but it is difficult to carry out the process by hand on cotton. Therefore, machines have been devised which will assist considerably in the process. These machines do not, however, completely eliminate the human factor. The three types of machine staplers are the Ball (or sledge) machine, the Baer, and the Suter-Webb. The Ball apparatus is applicable only to silver and requires a large sample. It is not used to any great extent in this country. The Suter-Webb is simply an elaboration of the Baer; in fact, two Baer instruments may be used instead of the Suter-Webb.

The Baer sorter consists essentially of a metal frame holding nine bottom combs, pointing upward, hinged at one end and all held by one pin at the other so that they may be dropped one at a time; in slots at the top are three top combs, pointing downward, which are removable. Accessories with the apparatus are a pair of wide-nosed tweezers, a wooden rake for pressing fibers into the combs, a needle for straightening fibers, and a transparent scale for reading the fiber diagram. The apparatus is used in the following manner for testing a sample of cotton:

1. Pull fibers from the sample as in hand stapling until a good-sized tuft, about 2½ inches long, is obtained. Straighten the fibers as much as possible but do not discard any. If the fibers are very dry, moisten them by breathing on them.

2. With the top combs removed and the sorter with back toward the operator, place the sample on the combs at the right side so that the fibers project about an inch from the needles. The end of the projecting tuft should look like the end of a water-color brush, i.e., bluntly tapering.

3. Using the tweezers, grasp the ends of the outermost fibers and draw them from the combs. Draw through the back comb to remove dirt or straighten.

4. Place these fibers on the combs at the opposite end of the sorter, with the tweezers against the nearest comb. Release the tweezers carefully and press the fibers into the pins with the wooden rake. The fibers should be in a narrow band.

5. Repeat this drawing, placing the fibers on this band, until all the projecting fibers from the first tuft are gone.

6. Put in the top combs, turn sorter around so that it now faces the operator, draw a straight chalk line as a base on a velvet-covered plate, as described in the section on hand stapling. If no fibers project from the nearest comb, lower the combs, one at a time, until fibers do project.

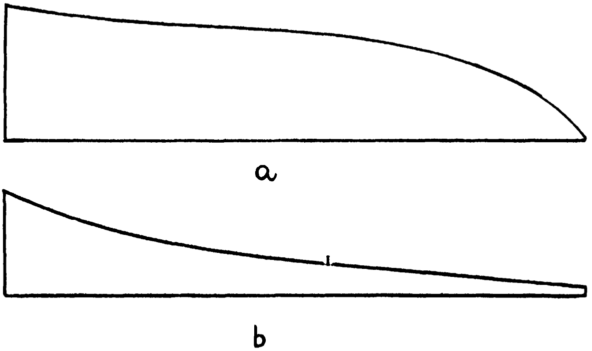

FIG. 6. Cotton Staple, Baer Sorter Diagram; (a) Comber Silver, (b) Comber Waste

7. With the tweezers, grasp the longest fibers near the ends and draw from the combs. Keep these fibers straight.

8. Place the lower front edge of the tweezers on the left end of the chalk line. If any fibers are not straight, use the needle to straighten them. Release the tweezers and withdraw them carefully, holding the fibers with the needle.

9. Repeat these operations, dropping the lower combs and removing the top combs as necessary. Place each new tuft of fibers at the right of those already drawn so that a uniform thickness of fibers is obtained. This is called an array.

10. Place the transparent scale on the array and read off the values desired, or make a tracing of the array, or weigh groups

of fibers as described in the stapling of wool. As an approximation, the average staple is the staple about one-quarter of the way along the array.

Figure 6 shows a tracing of a Baer diagram of comber silver and comber waste. Diagrams such as these are very valuable in analyzing the efficiency of textile machinery.

Hair Weight

Hair weight or, more correctly, hair weight per unit length is a means of expressing the fineness of a fiber. Various units have been used, such as ounces $\times 10^{-6}$ per inch, milligrams per inch, milligrams per 10 meters, and milligrams $\times 10^{-5}$ per centimeter: This last method of expression seems to be the most commonly used and the one to which reference will be made in this discussion. However, the A.S.T.M. uses micrograms per inch in reporting hair weights.

One of two methods may be used for determining fineness of fibers, measurement of diameter or measurement of hair weight. The measurement of diameter is done by a microscope and graduated wedge. This method is generally used at the present time in the determination of the quality or grade of wool but is not of much value in the case of cotton because of the fact that cotton is a hollow fiber and its properties depend not only on its width but also on the proportion of solid fiber to air space in that width. The hair-weight method, on the other hand, gives an accurate estimate of the amount of material in a fiber. This is much more nearly correlated to other fiber properties than diameter measurements of cotton. The disadvantages of the hair-weight method are the facts that the method is time consuming, requires considerable skill on the part of the operators, requires a microbalance, and gives an average result with no information as to the distribution of different sizes of fibers. Diameter measurements, then, are used principally on wool, and occasionally on cotton; hair weights are determined principally on cotton, occasionally on wool. As a matter of fact, hair weights are seldom obtained for routine work, even on cotton, but only in research work or in trouble-shooting.

As shown in Table VI, hair weights might be used to determine the quality or grade of wool provided that the fibers are not excessively medullated. However, diameter measurements are more convenient. Table VI also gives the hair weights for various types of cotton. There is considerable overlap, however, so that the method is not especially valuable for positively identifying a sample of cotton. The method will, however, show a difference between two cottons which might have the same staple. Hair weight will also give information as to the maturity of the cotton, a high hair weight indicating more mature cotton, a low hair weight of the same cotton indicating immaturity. Here again, however, more convenient microscopic methods are available for determining the maturity of cotton, e.g., the swelling method and the polarized-light method. A close correlation between the hair weight of cotton and the strength of the yarn spun from the cotton has been observed.

METHODS OF DETERMINING

There are three methods of determining hair weights: Clegg's method for use on cotton, Winson's method for use on wool, and the A.S.T.M. method which is used principally on cotton but can be used on any type of fiber.

Clegg's Method

Clegg's method consists of taking cotton, which has had the fibers more or less parallelized either by a machine sorter or by hand stapling, and cutting from the middle of the bundle of fibers with a special cutter consisting of a holder with two safety razor blades exactly 1 centimeter apart. About 200 pieces of hair are then weighed on a quartz fiber torsion microbalance. The zero of the balance must be checked often or the fibers must be weighed by the null method, that is, a one-milligram weight is placed on the balance, the deflection read, and enough fibers put on the balance to give the same deflection. The cutter should be tested at intervals by marking on a glass slide covered with wax and the distance between the marks should be measured. No special care is necessary to obtain average lengths of fiber as there is apparently very little correlation between fiber length and hair weight. Groups of fibers from the Baer diagram may be used for samples and the results averaged. Table VI gives Morton's results on various types of cotton.

TABLE VI
HAIR WEIGHTS OF FIBERS

	Wool		*Cotton*
Quality	*Hair weight per Centimeter at 13.6% regain milligrams* × 10^{-5}	*Variety*	*Hair weight per Centimeter milligrams* × 10^{-5}
80's	447	Egyptian	
70's	485	Sakel	113–151
64's	580	Assili	144–170
60's	690	Uppers	177–212
58's	771	American	
56's	919	Sea Island	102–136
50's	1123	Texas	167–211
48's	1260	Miscellaneous	178–235
46's	1290	South American	165–256
44's	1468	Indian	193–312
40's	1560	Indian-American	145–226
36's	1512	African-American	135–180
		American-Egyptian	167–175

Winson's Method

Winson's method, used on wool, consists in sampling the top as for staple, parallelizing the fibers by hand, and cutting a section out of the bundle of 3 or 4 centimeters in length, using a rule or, better, a metal gage. From this bundle, 500 to 700 fibers are counted out. They are rolled together in a small bundle, washed in two changes of benzene, rinsed in two changes of water, dried and weighed. A small sample (about 1 gram) of the original wool is put through the same treatments and the moisture determined on this sample. There is considerable correlation between the staple and the hair weight of wool, so that an average sample should be cut. The resulting hair weight is calculated to standard regain. Table VI gives Winson's values for the standard wool tops, recalculated to 13.6% regain and converted to milligrams × 10^{-5} per centimeter.

A.S.T.M. Method

The A.S.T.M. method consists in taking a number of fibers from an eighth-inch group of the Baer diagram, weighing, and calculating the hair weight. For example, if 100 fibers from 1 to $1\frac{1}{8}$ inches in length (average length = $\frac{17}{16}$ inches) weighed 0.54 milligrams, then the hair weight would be:

$$\frac{0.54 \times 1000}{\frac{17}{16} \times 100} = 5.1 \text{ micrograms per inch}$$

Resilience

Resilience is the springiness of a mass of fiber or the ability of the fiber to come back to its original volume after being compressed. This property is especially valuable in such fibers as cotton, kapok, and hair, which are used in mattresses. It is an important factor in the *feel*, *handle*, or *loftiness* of fabrics and must also be considered in such fabrics as necktie linings which are often crumpled. The resilience of fabrics will be taken up later when the stiffness and creasing properties of cloths are considered. At this time, the resilience of fiber masses will be discussed.

The principle of resilience measurement is the hysteresis of the load-volume curve. If we apply a load to a mass of fiber and measure the volume, we see that increasing load causes a decrease in volume. If we then unload the mass, we see that the volume increases, but does not follow the same curve as increasing load. The difference between the two curves is a measure of the resilience, a small difference indicating a high resilience and a large difference indicating a low resilience. Two methods have been devised for measuring resilience, the rubber-balloon method of Winson and the cylinder and plunger method described by Haven.

RUBBER-BALLOON METHOD

Winson's apparatus consists of a rubber balloon which holds the fiber and is enclosed in a glass vessel. Both the glass vessel and the balloon are connected to a mercury manometer and to a three-way stop-cock by means of which either one or both may be connected to a filter pump or to the atmosphere. The apparatus is first calibrated. It is then run with fiber in the balloon. The balloon and vessel are both evacuated, then air is admitted to the vessel but not to the balloon. This applies a load to the fiber mass which is measured by one manometer. The result is a decrease in volume which is measured by the other manometer. Winson used this method to measure the resilience of wools and found the results correlated well with expert judgment. Short fibers were found to be more resilient than long ones. A planimeter was used to measure the area of the hysteresis loops.

CYLINDER AND PLUNGER METHOD

Haven has described a method in use by the United States Federal Specifications Board in which 500 grams of fiber are placed in a hollow brass cylinder, 7½ inches in diameter, the axis standing vertically. A plunger is used to compress the material and a vernier is arranged on the screw driving the plunger so that the depth of the penetration may be measured. The screw is simply an inverted jack and the apparatus is coupled to a platform scale to read the load. The time element is important, since the volume will slowly change on application of a load. Therefore, readings are taken 2 minutes after application of the load. The resilience may be measured by the area of the hysteresis loop or, more simply, by observing the displacement on loading and unloading at a given low load with a high loading in between. Haven uses 50 pounds and 800 pounds as the two loads. On loading to 50 pounds, the reading is a; on loading to 800 pounds, the reading is b; on unloading to 50 pounds again, the reading is c. Then

$$\text{Resiliency factor} = \frac{c - b}{a - b} \times 100$$

Cloth resiliency may be measured in this instrument by cutting out discs, $7\frac{1}{2}$ inches in diameter, and separating them with discs of tin.

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Chapter 4

YARN TESTING

Counts and Denier

In determining the relative sizes of yarns, two systems immediately suggest themselves, measuring the diameter of the yarn and finding the weight-length ratio of the yarn. Measuring the diameter would be the more direct method but a little consideration will show that it is impractical for several reasons. One reason is that the edge of the yarn is not always sharp but very often fuzzy and indistinct. Another reason is that any instrument used would compress the yarn so that almost any value might result, depending upon the force applied.

Diameter of hard yarns may be measured by a gage similar to that discussed later for determining the thickness of cloth but the results are of questionable value. The diameters may be measured also by the microscope but even here the results vary, depending upon the tension used on the yarn. Proponents of the yarn-diameter measurement point out that it gives a better idea of the uniformity of the yarn but the same results may be obtained by weight-length ratios on short lengths of yarn. Diameter measurements, then, are seldom carried out and weight-length ratios are the usual methods of expressing the size of yarns.

WEIGHT-LENGTH RATIOS

Two systems of expressing weight-length ratios are in use:

1. Counts or number, i.e., the number of units of length in a unit of weight. This is used on all spun yarns except jute.*

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* Jute is an exception to these generalizations in that its number is given as *spyndles* which is the number of pounds in 14,400 yards of the jute yarn.

2. Denier, i.e., the number of units of weight in a unit of length. This is used on all continuous filament yarns.

In the counts system, the unit of length is a *hank* or standard skein and the unit of weight is the pound in the United States and Great Britain, 500 grams in France, and the kilogram in the rest of Europe.

For our purpose, we may define the counts as the number of hanks in a pound. The hank varies with the branch of the textile industry and also somewhat with geographical location. In cotton mills, for example, the standard hank is 840 yards; in worsted mills, the standard hank is 560 yards. Metric counts is the number of hanks of 1000 meters in 1 kilogram. French counts is the number of hanks of 1000 meters in 500 grams. As a result of the confusion caused by the use of all of these systems, it has been proposed by the A.S.T.M. that a universal system, called the *Typp* system be used for all fibers. The Typp system is based on a standard hank of 1000 yards as indicated by the name which consists of the initial letters of the phrase *thousands of yards per pound.* The length of the standard hank for various systems is given in Table VII.

The denier system was once in considerable confusion because different localities adopted different units of weight and length but, in this country at any rate, the adoption of the so-called *legal denier* has eliminated the confusion. The denier of a yarn is now expressed as the number of grams in a skein of 9000 meters. In English units, this is equal to the number of grains in 638 yards or the number of pounds in 4,464,483 yards.

Still another system of yarn numbering is the *Grex* system. This is based on the weight in grams of 10,000 meters and is suggested for adoption by all branches of the textile industry. It has no advantage over the denier system except the psychological one that none of the existing systems will be used and everyone will be inconvenienced equally. In England, the *GK*

Thus jute, although a spun yarn, is numbered in units of weight per unit of length.

system is being urged. This is based on grams per kilometer or 1000 meters.

Table VII
STANDARD HANKS OF YARN

Standard Weight = One Pound	
Typp system	1000 yards
Cotton, spun silk, spun rayon	840 yards
Worsted	560 yards
Woolen, cut system	300 yards
Woolen, run system	1600 yards
Flax, hemp	300 yards
Asbestos, glass	100 yards
Standard Weight = One Kilogram	
Metric system	1000 meters
Standard Weight = 500 Grams	
French system	1000 meters

Denier = grams per 9000 meters
= grains per 638 yards
= pounds per 4,464,483 yards
Grex = grams per 10,000 meters
= grains per 709 yards
Spyndle (jute) = pounds per 14,400 yards

Length Measurement

The length of the yarn is obtained by use of a yarn reel as illustrated in Fig. 7. This consists essentially of six arms of such a length that the perimeter of the hexagon formed by wrapping the yarn around the arms is 1 yard, 1½ yards, or 112.5 centimeters. The reel is operated by a crank or, in the most modern type, by a motor at constant speed. A revolution counter, calibrated in yards, is attached to measure the total length of the yarn reeled off and a bell is so connected as to give a warning ring just before a complete revolution of the counter. The yarn may be reeled off a cone, a spool, a bobbin, or a skein, passing over a pigtail which is used to obtain an approximately constant tension. The pigtails are moved sideways to spread the yarn out evenly on the reel. In order to obtain accurate results, three conditions are necessary:

1. The yarn must be spread out on the reel so that each revolution will reel off exactly the same length of yarn.
2. The tension must be constant and neither too much nor too little.
3. The speed of the reel must be constant.

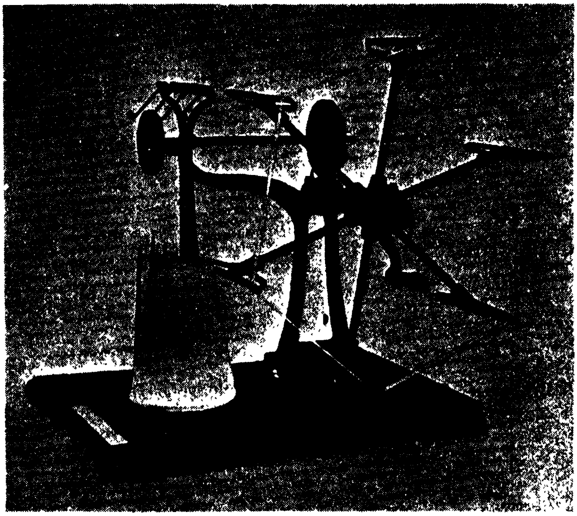

FIG. 7. Yarn Reel

In order that these conditions shall be met, the A.S.T.M. has proposed the following standard conditions for use of the reel:

1. A reel shall be used which spreads the yarn out on the reel and not more than 120 yards shall be reeled off in any one skein.
2. The tension shall consist of one full turn around the pigtail. This may be modified for loosely twisted yarns.
3. Standard speeds are specified for the reels as follows:
   a. Cotton yarns taken from bobbins or cones
      = 100 to 300 revolutions per minute
   b. Cotton yarns taken from parallel tubes or spools
      = 20 to 30 revolutions per minute

c. Rayon yarns taken from skeins
= 100 to 150 revolutions per minute
d. Rayon yarns taken from spools, cops, tubes, or cones
= 200 to 300 revolutions per minute
e. Woolen and worsted yarns
= no standard speed given

The 1½ yard reel is now the most frequently used, having replaced the one-yard reel almost entirely. For silk or rayon, the 112.5 centimeter reel is used because 200 turns will give a skein of 225 meters which is an even subdivision $\frac{1}{40}$ of 9000 meters, the standard hank. The 1½ yard reel may, however, be used to measure the denier of silk or rayon by using the proper conversion factor. Shorter lengths of yarn may be used for counts or denier determinations if measured under standard tension, which is enough to straighten but not stretch the yarn. This will be discussed later in the section on crimp.

The reel may also be used to check the total length of yarn on a package by passing the yarn three times around the reel and winding it up on a drum until the whole package is unreeled. The total yardage of the package is then indicated by the number of revolutions of the reel.

Weight Measurement

Weighings of the yarn may be made by:

1. Grain scales at room conditions
2. Analytical balance at room conditions
3. Special quadrant scales at room conditions, usually direct reading
4. Torsion balance and drying oven
5. Analytical balance and drying oven

Weighing oven-dry in the oven by means of a balance mounted on the oven is the most accurate method of getting the true weight. However, it is more convenient to weigh at room conditions, provided that standard conditions of temperature and relative humidity are maintained in the room.

The grain scales are in general use for determining the weight of skeins of yarn at room conditions. These are calibrated in grains, and a simple calculation gives the counts of the yarn if the standard skein is weighed. The analytical balance gives more accuracy than is usually necessary and is valuable principally in determining counts or denier from small lengths of yarn, as unraveled from a cloth for example. The special quadrant scales are usually direct reading, provided that a certain definite length of yarn is taken. These are for plant use only; they are not accurate enough for precise determinations.

A more accurate method is to weigh the skeins when they are oven-dry. The so-called *conditioning ovens*, usually supplied to the textile industry, consist of an oven with a torsion balance accurate to 1 centigram or to $\frac{1}{10}$ grain mounted on it. These are sufficiently accurate for counts determinations, but a combined oven and analytical balance is even more accurate besides being useful in getting counts of small pieces of yarn and for various chemical textile tests.

Calculation of Counts and Denier

The formulae for calculating counts and denier from length and weight determinations are as follows:

$$N = \frac{L}{W} \times \frac{7000}{H}$$

where

N = counts or number
L = length in yards
W = weight at standard regain in grains
H = length of standard hank in yards (Table VII)

$$D = \frac{9000}{L'} \times W' \qquad \text{or} \qquad D = \frac{638}{L} \times W$$

where

D = legal denier
L' = length in meters
W' = weight at standard regain in grams

The use of these formulae may best be shown by illustrative examples. Suppose that a skein of 120 yards weighs 25 grains at standard conditions, then

$$N = \frac{L}{W} \times \frac{7000}{H} = \frac{120}{25} \times \frac{7000}{840} = 40$$

The yarn counts is expressed as a figure with *'s* and, preferably, with some phrase which will give the system used. The above result would be expressed as 40's cotton count.

A dry spun worsted skein of 80 yards weighs 50 grains when oven-dried; what is the worsted count? Here, W is not 50 grains but 50 grains plus 15.0% (Table IV) or

$$W = 50 \times 1.15 = 57.5 \text{ grains}$$
$$N = \frac{80}{57.5} \times \frac{7000}{560} = 17.4\text{'s worsted}$$

A woolen yarn skein of 100 yards weighs 3.25 grams when oven-dried; what is the Typp number? Here, we must convert from grams to grains (Table VIII) and then add the standard regain (Table IV).

$$W = 3.25 \times 15.43 \times 1.13 = 56.6 \text{ grains}$$
$$N = \frac{100}{56.6} \times \frac{7000}{1000} = 12.3\text{'s Typp}$$

TABLE VIII
ENGLISH AND METRIC EQUIVALENTS

1 yard = 36 inches = 0.9144 meters
1 meter = 39.37 inches = 1.094 yards
1 pound = 7000 grains = 453.6 grams
1 ounce = 437.5 grains = 28.35 grams
1 gram = 15.43 grains

A viscose rayon skein of 225 meters weighs 2.30 grams when oven-dry; what is the denier?

$$W' = 2.30 \times 1.11 = 2.55 \text{ grams}$$
$$D = \tfrac{9000}{225} \times 2.55 = 102\text{d}$$

Here, the denier is indicated by the d after the figure.

A silk skein of 120 yards weighs 2.75 grams when over-dry; what is the denier? Here we have one measurement in English units and one in metric units; we must convert one to the other, so the calculation may be done in one of two ways:

$$L' = 120 \times 0.914 = 109.5 \text{ meters (Table VIII)}$$
$$W' = 2.75 \times 1.11 = 3.05 \text{ grams}$$
$$D = \frac{9000}{L'} \times W' = \frac{9000}{109.5} \times 3.05 = 250\text{d}$$

or

$$L = 120 \text{ yards}$$
$$W = 2.75 \times 15.4 \times 1.11 = 47.0 \text{ grains}$$
$$D = \frac{638}{L} \times W = \frac{638}{120} \times 47.0 = 250\text{d}$$

These examples are elementary ones, only; more complicated examples will be given later when the effect of combining two or more yarns will be taken up.

When fabrics are dissected to obtain the counts of the constituent yarns, it is sometimes necessary to weigh very small lengths of yarn, after the yarns have been straightened and measured. An analytical balance is generally used for this purpose and the following formulae may be applied:

$$N = \frac{I}{M} \times \frac{12{,}600}{H}$$
$$D = \frac{M}{I} \times 354$$

where

I = length of yarn in inches

M = weight at standard regain in milligrams

The A.S.T.M. has specified tolerances on counts and deniers. Tolerances are the amounts above and below the specified value which the average of a lot of yarn may vary and still be acceptable. These tolerances are given in Table IX.

TABLE IX
YARN AND FABRIC TOLERANCES
Yarns

Counts and Denier	%
Cotton	3.0
Spun rayon	5.0
Flax, hemp, ramie	10.0
Woolen, coarser than No. 3 Typp	10.0
No. 3 to No. 6 Typp	7.5
No. 6 and finer	5.0
plied yarns	5.0
Worsted, coarser than No. 12 Typp	5.0
No. 12 to No. 17	3.5
No. 17 to No. 23	3.0
No. 23 and finer	2.5
plied yarns	2.5
Rayon, singles, 150d or coarser	6.0
Singles, finer than 150d	8.0
Plied, 150d or coarser	8.0
Plied, finer than 150d	10.0
Jute, 12 pounds per spyndle and over	5.0
8 to 12 pounds per spyndle	7.5
Under 8 pounds per spyndle	10.0
Twist	
Cotton	5.0
Spun rayon	5.0
Jute	5.0
Flax, hemp, ramie	10.0
Woolen and worsted	7.5
Rayon, under 7½ TPI	20.0
7½ to 10 TPI	10.0
Over 10 TPI	5.0

Knit Goods

Wales per inch, courses per inch	5.0
Width	3.5
Weight	5.0

Cloth

Number of yarns per inch, warp	−3 to +4
Filling	−5 to +7

Plies and Filaments

Under this heading, we shall consider the number of units which go to make up a yarn. When a rayon is spun, a liquid is forced through a die containing a certain number of holes. Similarly, when a real silk yarn is made, it is composed of a certain number of cocoons which are unwrapped and combined to form the silk yarn. It is obvious that if we have two yarns of the same denier but one contains more filaments than the second, then the first yarn will be more pliable and flexible and softer than the second. In rayon yarns, then, it is customary to give the number of filaments as well as the denier; thus, a 150-denier yarn composed of 40 filaments would be given as 150d 40f or as 150/40.

If the filaments are fairly coarse (3-4 denier per filament or more), the easiest way to count the filaments is to cut a piece about an inch long, untwist the yarn if any appreciable twist is present, and withdraw the filaments from the bundle one at a time by means of a pair of tweezers, counting the filaments as they are withdrawn. If the filaments are fine but not too numerous, a mount may be made and the filaments counted through a binocular microscope. If the filaments are fine and numerous, it is safest to stain the rayon, make a wax candle, cut cross-sections, and count the number of cross-sections in the mount, using a microscope.

In the case of the spun yarns, the basis is the singles yarn which is made up of a large number of individual fibers held together by a certain amount of twist. If two or more singles yarns are twisted together, we have a ply yarn and the number of plies is indicated when giving the counts. Thus, 40/2 or 2/40 means that two 40's singles yarns have been twisted together to give a ply yarn. It is spoken of as *forties two ply* or as *two ply forties*. Some localities use one phrase, and other localities use the other. When two or more ply yarns are twisted together, the result is known as a cord. Thus, 23/5/3 is a tire cord composed of five 23's yarns plied together, and three of these plies twisted together to form the cord. It is referred to as a *twenty-threes, five ply, three cord.*

One other type of yarn should be mentioned at this time. It is now fairly common in cheaper goods to obtain the strength of a cotton yarn combined with the appearance and feel of a woolen yarn by twisting woolen fibers about a cotton yarn in such a way as to completely cover the yarn. This is known as a *core yarn* and the counts is usually figured in the woolen or worsted system according to the yarn which it is imitating and replacing. This is not really a ply yarn, since the two types of fibers are not twisted about each other but only the woolen is twisted about the cotton.

If we had a two-ply cotton yarn of which 120 yards weighed 50 grains at standard conditions, then according to previous methods of calculation, the counts would be:

$$N = \frac{L}{W} \times \frac{7000}{H} = \frac{120}{50} \times \frac{7000}{840} = 20\text{'s}$$

The yarn, however, is not really a 20's yarn but is roughly two 40's yarns twisted together. The figure (20's) obtained in the above manner, in the case of a ply or cord yarn, is referred to as the *equivalent singles counts*, or simply as the *equivalent counts.* To obtain the true counts, it is necessary to know how much the yarn shortened in length during the plying operation; this figure is known as the *twist contraction* and its determination will be given later. At this time, we shall give only the formula by means of which it is calculated:

$$C = \frac{F - L}{F} \times 100 \qquad \text{or} \qquad F = \frac{L}{1 - \frac{C}{100}}$$

where

C = twist contraction in per cent
F = original length of the singles yarn before plying
L = length of the ply yarn

Then, in the example given above, if the twist contraction was 3%:

$$F = \frac{120}{1 - \frac{3}{100}} = \frac{120}{0.97} = 123.8 \text{ yards}$$

Then, since the length of the original singles yarn was 123.8 yards and the weight was half of 50 grains, or 25 grains:

$$N = \frac{L}{W} \times \frac{7000}{H} = \frac{123.8}{25} \times \frac{7000}{840} = 41.2$$

The yarn then is not a 20's yarn, but a 41.2/2 or 2/41.2's. This is known as the *true counts* as opposed to *equivalent counts* and involves twist contraction as well as weight and length. The following examples will show the methods of calculation in other cases:

A worsted two-ply yarn, 80 yards in length, weighs 1.95 grams when oven-dry and the twist contraction is 5%. What is the equivalent counts of the yarn, and what is the true counts?

$$W = 1.95 \times 15.4 \times 1.13 = 34.0 \text{ grains}$$

$$\text{Equivalent counts} = \frac{80}{34.0} \times \frac{7000}{560} = 29.4\text{'s}$$

$$F = \frac{80}{1 - \frac{5}{100}} = \frac{80}{0.95} = 84.2 \text{ yards}$$

$$\text{True counts} = \frac{84.2}{17.0} \times \frac{7000}{560} = 62.0$$

The yarn is 62/2 or 2/62's worsted.

A cotton cord is composed of three ply yarns twisted together with a twist contraction of 6%. Each of the ply yarns is composed of two singles yarns twisted together with a twist contraction of 2%. When oven-dry, 120 yards of the cord weighed 18.0 grams. What is the true counts of the cord?

$$W = \frac{1}{6} \times 18.0 \times 15.4 \times 1.075 = 49.4 \text{ grains}$$

Length of ply yarn before cording

$$= \frac{120}{1 - \frac{6}{100}} = \frac{120}{0.94} = 127.7 \text{ yards}$$

Length of single yarn before plying

$$= \frac{127.7}{1 - \frac{2}{100}} = \frac{127.7}{.98} = 130.2 \text{ yards}$$

Then,

$$N = \frac{L}{W} \times \frac{7000}{H} = \frac{130.2}{49.4} \times \frac{7000}{840} = 22.0$$

The cord is 22/2/3 cotton.

If the equivalent counts is known or has been determined, the following formulae may be used to determine the true counts of ply yarns or cord yarns:

Ply yarns

$$\text{True counts} = \frac{n}{1 - \frac{C}{100}} \times N$$

Cord yarns

$$\text{True counts} = \frac{n_1 n_2}{\left(1 - \frac{C_1}{100}\right)\left(1 - \frac{C_2}{100}\right)} \times N$$

where

n = number of singles in ply or plies in cord.

For many purposes, the twist contraction may be neglected and an approximation is obtained which is satisfactory for the purpose.

Twist

Twist is the measure of the spiral turns given to a yarn in order to hold the constituent fibers or threads together. Up to a certain point, an increase in the twist tends to make a stronger yarn; but above this point, the additional twist adds a strain to the fibers which results in a weaker yarn. In general, then, there is a certain limited range of twist which is desirable. For certain effects, however, less twist (soft twist) or more twist (hard twist) may be necessary. According to the kind and amount of twist, yarns may be classified as:

1. Singles yarn (see preceding section)
2. Ply yarn (see preceding section)
3. Cord (see preceding section)
    - a. Hawser twist
    - b. Cable twist
4. Core yarn (see preceding section)
5. Crepe yarn, i.e., yarn with a large amount of twist, usually held in place by a coating of gelatin size
6. Novelty yarn, i.e., yarn of nonuniform twist where strength is sacrificed for appearance

MEASURING TWIST

Two general methods of measuring twist are available: (1) a microscopic method, depending upon the measurement of the angle of spirality (helix angle), which will not be taken up at this point; (2) a method using a *twist counter* which untwists a given length of yarn and measures the number of turns necessary.

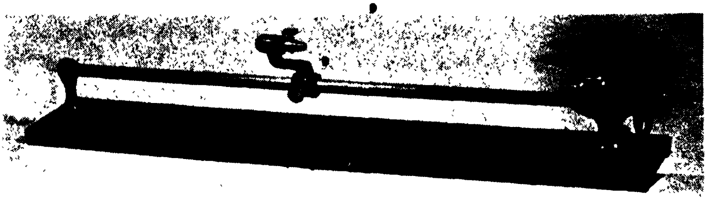

FIG. 8. Simple Twist Counter. (*Courtesy of Scott Testers, Inc.*)

Reduced to fundamentals, the twist counter consists of two jaws, one nonrotating but movable on a rod, so that the distance between the jaws may be varied, the other jaw fixed in position but capable of rotation by means of a handle and having a revolution counter attached to read the number of turns.

In taking the yarn from the packages to test for twist, the yarn should be drawn off from the side of the package and not over the top, otherwise an additional twist would be added to or taken away from the yarn. The yarn should be tested in successive lengths with only an inch or less between samples, so that a record of the variation of the yarn will be obtained. The yarn is untwisted by the rotating jaw and the number of turns of the jaw divided by the distance between the jaws (gage length) gives the *turns per inch*, usually abbreviated and referred to as TPI. The simple twist counter (Fig. 8) is used as follows:

1. The gage length is set at 10 inches and the ply yarn clamped in the jaws; the yarn should be straight but not stretched. The revolution counter should be at zero.
2. The rotating jaw is revolved until all the ply twist is removed and the strands are parallel, so that a needle may be passed between them from one jaw to the other.
3. The number of turns of the rotating jaw, as shown by the dial, is recorded. This value divided by ten (gage length) is the TPI of the ply yarn. Since the dial usually reads only up to 100 revolutions, a note should be made of the number of complete revolutions of the dial.
4. With a sharp razor blade or scissors, all but one of the singles yarns are cut from between the jaws.
5. Holding the left end of the single yarn so that it will neither twist nor untwist, the nonrotating jaw is moved up to the correct gage length for the singles yarn, which is then clamped in place. The gage length for singles yarns is 1 inch for cotton and 4 inches for woolen and worsted yarns.
6. The singles yarn is untwisted by rotation of the revolving jaw until the fibers are as nearly parallel as possible. A magnifying glass helps to determine when the fibers are parallel in a cotton yarn. In the case of woolen and worsted yarns, a needle may be used as for ply yarns.
7. The number of turns registered on the dial is read again and divided by the gage length to obtain the TPI of the singles yarn.

A convenient method of recording and calculating results follows:

	Ply Twist		*Single Twist*	
Balance	(see later)			
Direction of twist	(see later)		(see later)	
Gage length	10 inches		4 inches	
Twist	110 turns =	11.0 TPI	24 turns =	6.0 TPI
	114	11.4	25	6.3
	96	9.6	22	5.5
	106	10.6	24	6.0
	110	11.0	27	6.8
	Total	53.6		30.6
	Average	10.7 TPI		6.1 TPI

As shown above, at least five determinations, preferably ten, should be made on each sample. In testing a shipment of yarn, at least five samples, preferably more, should be taken. The results may be calculated according to the methods in Chapter 1 to get true value and variation of the yarn.

The simple twist counter gives results accurate enough for all ordinary purposes, but the following points should be noted:

1. The tension on the yarn when placed between the jaws is a variable quantity. Great tension would result in fewer TPI; less tension would result in more TPI.

2. In the case of singles cotton yarns particularly, it is difficult to decide just when the fibers are parallel, since opposite twist may start to go into some fibers before others are wholly untwisted.

3. On untwisting a ply yarn, the yarn slackens between the jaws due to the removal of the twist. This expansion of the yarn is equal to the twist contraction (mentioned in the section on plies) and should be measured if the true counts of the yarn is to be determined.

The more elaborate twist counter, shown in Figure 9, takes the above points into consideration. This instrument is similar in principle, although usually larger in size, to the simple type with the exception that the nonrotating jaw is capable of sliding in a sleeve and has a constant-tension device on which the tension may be varied by means of weights. The sliding arm is calibrated so that twist contraction may be read. It also has a feeler arm which may be rested on the midpoint of the singles yarn being untwisted with any desired weight. It is used as follows:

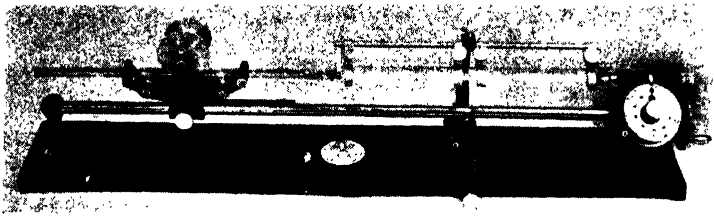

FIG. 9. Accurate Twist Counter with Take-up Device. (*Courtesy of Scott Testers, Inc.*)

1. With the sliding arm clamped at zero, the revolution counter at zero, and a gage length of 10 inches, place the ply yarn between the jaws.
2. On the constant-tension device place weights to give the following tensions in grams:

Cotton = 3 grams (on the feeler)

$$\text{Spun rayon} = \frac{131}{\text{Typp number}}$$

Woolen and worsted yarn tension = weight of 100 yards of yarn

Rayon yarn tension = 10 grams (for 75d or finer)
20 grams (75d to 150d)
30 grams (coarser than 150d)

3. Release the clamp on the sliding arm and read the extension.
4. Remove the ply twist, read the extension and the revolution counter; divide by the gage length.

5a. Woolen and Worsted Yarns

Clamp the sliding arm at zero again. Cut out all singles yarns except one. Set to a gage length of 4 inches, holding the twist in the singles yarn meanwhile.

5b. Cotton and Spun Rayon Yarns

Clamp the sliding arm at zero again. Cut out all singles yarns except one. Set to the gage length desired (at least 5 inches, preferably 10 inches). Place the feeler arm at the midpoint of the yarn and resting on it, set the weight on the feeler arm at 3 grams. Loosen the rotating jaw and tighten the yarn until the feeler arm is $\frac{1}{8}$ inch below the straight level of the yarn. Clamp the yarn again in this position.

6a. Woolen and Worsted Yarns

Untwist the singles yarn until a needle can be run from one jaw to the other. Read the dial and divide by the gage length.

6b. Cotton and Spun Rayon Yarns

Remove the feeler arm temporarily. Untwist the singles and continue to retwist in the opposite direction. Replace the feeler arm and twist until the feeler arm returns to its original position. Read the dial and divide by twice the gage length.

An example of the data and calculations for the more accurate twist counter is shown in the table on page 64.

The per cent twist contraction is calculated from the formula previously given:

$$C = \frac{F - L}{F} \times 100$$

where

C = per cent twist contraction
F = original length of the singles yarn before plying
L = length of the ply yarn

In the above case, the calculation is:

$L = 10$ inches
$F = 10 + 0.235 = 10.235$ inches
$F - L = 0.235$ inches

then,

$$C = \frac{0.235}{10.235} \times 100 = 2.30\%$$

Note that the formula for twist contraction may be expressed as:

$$C = \frac{100 \times \text{extension}}{\text{gage length} + \text{extension}}$$

A simpler method of determining the twist in singles yarns is as follows:

1. With the revolution counter at zero, set the jaws for a separation of 5 inches. Attach 50 grams to the tension device. Insert the singles yarn. Release the tension device and read the length L_1.

	Ply Twist			*Single Twist*	
Balance	(see later)				
Direction	(see later)			(see later)	
Gage length	10 inches			5 inches	
Twist	Turns	TPI	Extension	Turns	TPI
	168	16.8	0.20	90	9.0
	170	17.0	0.25	80	8.0
	165	16.5	0.23	100	10.0
	166	16.6	0.25	90	9.0
	172	17.2	0.25	85	8.5
Total		84.1	1.18		44.5
Average		16.8	0.235		8.9

2. Tighten the set screw so that the length remains fixed and turn the rotating jaw to add twist until the yarn breaks. Read the number of turns, N_1.
3. Put a second sample in the apparatus in exactly the same way. Read the length L_2, and lock in position.
4. Turn the rotating jaw in the opposite direction so as to first untwist the yarn and then to twist it in the opposite direction until it breaks. Read the number of turns, N_2.

5. Calculate the TPI from the formula:

$$TPI = \frac{N_2}{2L_2} - \frac{N_1}{2L_1}$$

These more accurate twist counters may also be obtained with a motor-driven revolving jaw, a rather unnecessary refinement, since the time required to untwist a sample is small compared to the time required for adjusting tensions, setting gage lengths, and recording data.

The A.S.T.M. tolerances on twist are given in Table IX.

Direction of Twist

In describing the direction of twist, there was for years much confusion since it was referred to as *right-hand* or *left-hand* twist. Some branches of the industry described as *right-hand* yarn that yarn which was twisted by turning to the right, while other branches described as right-hand yarn that which was untwisted by turning to the right. The A.S.T.M. method of describing direction of twist is to be preferred. This system

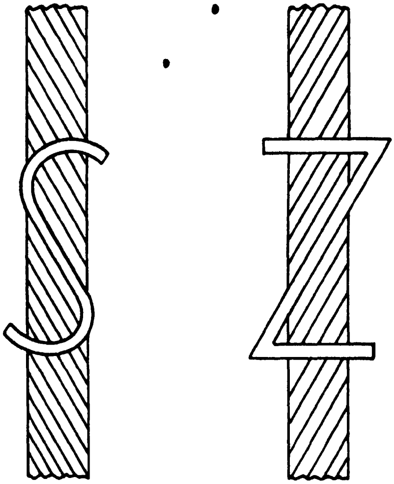

FIG. 10. "S" and "Z" Twist

describes the twist as S or Z according to the following definition: "a yarn or cord has S twist if, when held in a vertical position, the spirals conform in slope to the central portion of the letter S, and Z twist if the spirals conform in slope to the central portion of the letter Z." Figure 10 illustrates both types of twist.

In plying yarns, the ply twist is usually, although not always, opposite to the singles twist. Cords are of two types: hawser twist and cable twist. Hawser twist has the singles twist and ply twist in the same direction and the cord twist in the opposite direction; tire cords, for example, have the singles yarns of Z twist, ply yarns of Z twist, and cords of S twist. Cable twist has the ply twist opposite to the singles twist, and cord twist opposite to ply twist; a cabled cord, for example, might have singles yarns of Z twist, ply yarns of S twist, and cords of Z twist.

Balanced Twist

A yarn is said to be in balance if the amount of twist is just enough to keep the component yarns or fibers in position. If

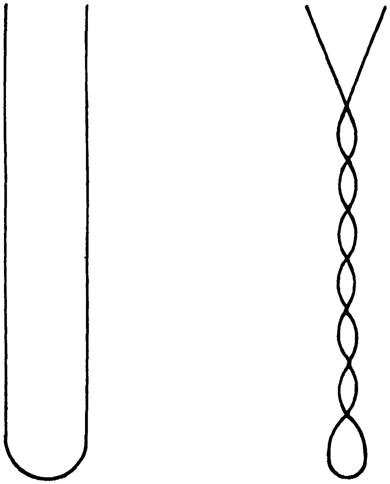

FIG. 11. Balanced and Unbalanced Yarns

more twist than this is put in, the yarn will kink and snarl; if less twist is put in, the yarn will tend to untwist and again will kink. For knitting machines and sewing machines especially, the question of balance in the yarn used is very important. A yarn is tested for balance by taking a one-yard length in the hands, each end held by thumb and forefinger of one hand, and bringing the ends together. If the yarn hangs in a loop without twisting about itself, it is in perfect balance; if it twists about itself, it is not balanced. A rough indication of unbalance is obtained by noting the number of times the yarn twists about itself, but usually a yarn is reported simply as balanced or not balanced. Figure 11 illustrates balanced and unbalanced yarns.

Corkscrew Twist

In making a ply yarn, all the singles yarns should twist about each other equally, so that when a load is placed on the yarn each strand will take its share of the load; if, however, one yarn is straight and the others are twisted about it, applied load will all be taken up by the straight yarn until it snaps. Then the load will be applied to the others. This type of twist is known as *corkscrew twist* and is shown in Fig. 12. Corkscrew, while generally undesirable, is sometimes obtained deliberately for the manufacture of novelty yarns.

There are several causes of corkscrew. If one yarn is under greater tension than the others, it will tend to be straight while the others corkscrew about it. If one yarn is fed straight into the twister while others come in at an angle, corkscrew will result. The number of plies in a yarn may determine corkscrew. Thus, a five-ply yarn has little tendency to corkscrew, the five yarns arranging themselves on the circumference of a circle. A six-ply yarn, however, will nearly always corkscrew since one of the plies is forced into the center of the circle of yarns. It is for this reason that tire cords, where a maximum of strength is required, are made five ply. In general, three, five, and eleven-ply yarns seem to have the least tendency to corkscrew. Size of yarns also is a cause of corkscrew. Combining a thin and a thick yarn nearly always results in a corkscrewed yarn.

Corkscrew is measured by taking a long length of yarn, attaching a small weight to each ply (to give constant tension), and untwisting the yarns. The length of each ply is measured, the average length obtained, and the deviation of each length from the average is calculated. The sum of the deviations expressed as percentage of the average length is the per cent corkscrew of the yarn. Obviously, if there were no corkscrew all the lengths would be the same, the deviations and the per cent corkscrew would then be equal to zero. Haven has described a convenient apparatus for this determination. However, for only occasional determinations, a yardstick and a number of small spring paper clamps may be used, the paper clamps acting as the tension weights.

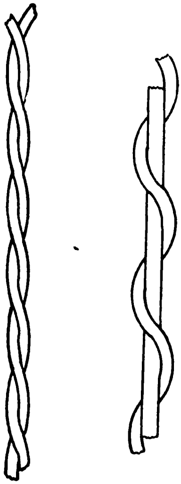

FIG. 12. Ordinary and Corkscrew Twists

Twist Factor

As indicated in the introductory paragraph of this section the maximum strength of a given counts of yarn is obtained with a definite amount of twist. This relationship is shown graphically in Fig. 13. The usual method of expressing the relationship between counts and twist is by the formula:

$$\text{TPI} = \text{K}\sqrt{\text{N}}$$

where

TPI = turns per inch
N = counts of the singles yarn
K = twist factor

It has been established by experiments and practice that the maximum strength of a yarn is obtained for a definite value of K. In the case of ring-spun cotton yarns, for example, the following values of K have been found to give the best results.

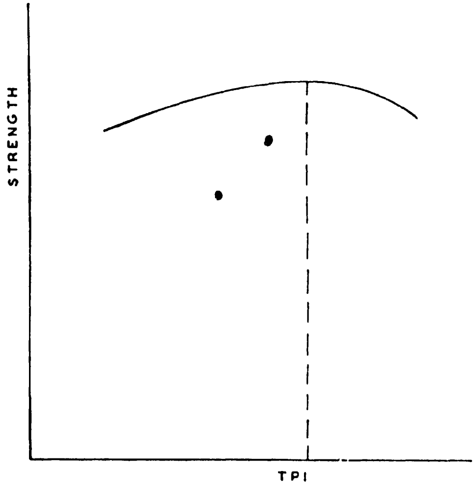

FIG. 13. Relation between Strength and Twist of Yarns

Warp yarns, 35's and less	4.75
Warp yarns, 35's to 80's	4.50
Warp yarns, 80's to 110's	4.25
Filling yarns, medium numbers	3.50

Filling yarns usually have a lower twist factor because strength is not so important and appearance is more important.

Ply yarns frequently have twist factors of 4 to 6, while crepe yarns are often two or three time ordinary yarns.

Nomenclature

The A.S.T.M. has sponsored a system of nomenclature for yarns which expresses not only the counts or denier but also the twist. This system may be best shown by a series of examples:

28S16 cotton means a singles cotton yarn of 28's count and with 16 turns per inch of S twist.

125-90S3 acetate means an acetate yarn of 125 denier and containing 90 filaments which have been given 3 turns of S twist.

23Z16/5S11 cotton means a five-ply cotton yarn; the singles are 23's with 16 turns of Z twist and the ply twist is 11 turns of S twist.

23S20/5S15/3Z12 cotton means a cord made up of singles which are 23's with 20 turns of S twist; five of these singles are twisted with 15 turns of S twist into a ply yarn and three of these ply yarns are twisted with 12 turns of Z twist into a cord.

Seriplane and Regularity

Methods for determining the regularity, uniformity, or evenness of yarn samples may be classified as follows:

1. Statistical methods
   a. Counts
   b. Twist
   c. Thickness
   d. Strength
2. Visual inspection, seriplane or inspection boards

The use of statistical methods depends upon making a large number of physical tests of one kind, such as counts or twist, on a large number of small samples; the results are then computed as discussed in Chapter 1 so as to determine the per cent variation. Thickness has also been used for this purpose, methods having been developed for measuring the thickness of yarns by recording feeler gages of various types. The use of the microscope for measuring the width of yarns has also been suggested. All of these methods, however, simply give the uniformity of the yarn in respect to only one variable. For example, the yarn might be very uniform in respect to counts but the twist might be very uneven. Strength tests are better in this respect and will be discussed later.

The most common way of comparing yarns for uniformity is by means of the inspection board or *seriplane*. The seriplane was originally devised for use in the silk industry but has been extended to other textile branches. It consists essentially of a device for winding yarn at even intervals around a board of a color contrasting with the yarn. The seriplane, in its simplest form, winds yarn from one or two packages on a black board. Machines are made, however, which wind a large number of boards at a time. After winding, the board is viewed at a little distance for general evenness or density of yarn, being observed especially for streaks. The boards are then examined more closely for:

1. Thick and thin places
2. Slubs, i.e., bunches of loose fiber caught in the yarn
3. Uneven twist
4. Corkscrew twist
5. Oil or rust spots
6. Hairiness, i.e., excessive number of protruding fibers
7. Broken filaments, in the case of silk or rayon

The test is, in general, qualitative but it may be made roughly quantitative by counting the number of imperfections per board. Permanent records may be made by photographing the boards, to be compared later with other photographs. Standard photographs of yarns are obtainable from the A.S.T.M. If 80% or more of the boards are equal to or better than the standard, the yarn is considered acceptable.

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Chapter 5

FABRIC TESTING—CONSTRUCTION

Types of Fabrics

Before taking up the various tests usually given fabrics, it will be necessary to discuss briefly the various types of fabrics from the point of view of methods of manufacture, production of pattern, general properties of the finished product, and some of the terms used in referring to the fabric. These will be discussed only in a very elementary way because we are concerned primarily with the testing and not with production of the fabrics. Practical experience and the study of other texts are necessary to supplement this very elementary survey.

The textile fabrics may be classified as follows:

1. Felts. These are fabrics made by uniting loose fibers into a uniform sheet.
2. Knit goods. These are fabrics made by looping one yarn about itself.
3. Braids and laces. These are fabrics made by twisting many yarns about each other.
4. Woven goods. These are fabrics made by passing one set of yarns above and below another set of yarns at right angles.

    a. Tapes and ribbons. These are made very narrow and on special looms.
    b. Mechanical fabrics. These are ordinary wide goods of fairly simple weave where appearance is secondary to strength or other physical properties.
    c. Dress goods, suitings, coatings, poplins, broadcloth, etc. These are fabrics of fairly simple weave but appearance is more important than in the other fabrics.

d. Print goods. These are of fairly simple weave but with a complicated color pattern applied by a printing process.
e. Tapestries and other similar fabrics. These are of very complicated weave produced by complex looms or Jacquard looms. Appearance is usually the most important quality in these goods.
f. Pile fabrics such as plushes, velvets, corduroys, and carpets. These are fabrics in which some of the yarns are cut during or after weaving so that the cut ends form the face of the fabric.
g. Multi-ply fabrics such as some collar materials. These are fabrics consisting of two separate warp and filling fabrics, united by another set of warp yarns which are common to both layers.

FELTS

Felts have no regular structure but consist of masses of fiber (usually hair fibers) united together by chemical treatments. Tests made on felt are usually for content and are chemical and optical tests. Thickness is the most important physical test. Porosity is sometimes determined.

KNIT GOODS

Knit goods consist of a series of loops of a single yarn; other yarns may be used to make stripes but usually only one strand at a time. The lengthwise loops of the fabric are known as wales and the crosswise loops as courses. Knit goods are often made in tubular form so that there are no edges. As hosiery and underwear form the bulk of the knit goods with their use as dress goods increasing, in general, appearance is the most important property. Physical tests, then, are not so important on knit goods as they come on the market; most of the testing of knit goods is done in the process of manufacture and carried out on the yarn which goes into the goods.

BRAIDS AND LACES

Braids and laces, also, are used principally for ornamentation and again the most important tests are those made upon the constituent yarns. This class of fabrics is, in point of quantity, relatively unimportant.

WOVEN GOODS

Woven goods are by far the most important group of fabrics. They are made in a loom by arranging a number of yarns (the warp) under tension and capable of being lifted and dropped by the harness mechanism, each individual yarn of the warp (end) passing between a set of combs called the reed. With some of the ends lifted and some dropped to form an angle (shed), a shuttle flies across carrying one yarn (pick); these cross yarns are known as the filling. Different patterns are made by putting different yarns in the warp and in the filling, also by lifting the warp yarns in various combinations. The filling yarns change direction at each edge of the fabric and to keep them firmly in position at the edges, a special lot of warp yarns very close together (the selvedge) is put in. Obviously, the cloth is firmer and, in general, different in properties near the selvedge, so it is a general rule that no physical tests shall be made nearer than one-tenth the width of the fabric to the selvedge. The use to which the fabric is to be put governs the tests which are given it. In any case, an analysis of the constituent yarns and their arrangement in the fabric should be made, preferably by an experienced designer. The tests which we shall discuss are given under the assumption that the design of the cloth has already been passed on and found to be correct.

Mechanical fabrics are fabrics to be used where appearance is unimportant. Usually, strength is most important, but sometimes other properties are equally or more desirable. In airplane wing fabric or balloon cloth, the permeability is an important factor which, in turn, is dependent upon the size and closeness of the yarns in the cloth. In bandage cloth or toweling, the ability to absorb liquids is very important; in canvas and umbrella cloth, waterproofness is most desirable.

In cloth to be used for clothing, both strength and wearing properties are important but must be combined with a good appearance; good draping qualities, a pleasant *handle*, and preferably a light weight. Other properties which may be desirable in special clothing are waterproofness, coolness or warmth, or ability to resist creasing.

In pile fabrics, in addition to appearance and construction, the most important physical property of the cloth is the ability of the cut ends or pile to remain upright under a load or, if bent by the load, to regain its upright position when the load is removed. This involves the study of the thickness of the fabric under varying loads and the resiliency of the fabric.

To study every physical property of every known fabric would be out of the question. We shall, therefore, take up only the more important properties which should, however, suggest a method of approach to the determination of other properties.

Fabric Count

This determination measures the number of warp yarns per linear inch (ends per inch or *sley*) and the number of filling yarns per linear inch (picks per inch or *shot*). In the case of knit goods, the corresponding values are wales per inch and courses per inch.

There are four general methods of determining fabric count:

1. Unraveling the cloth
2. Use of the pick glass
3. Mechanical pick counter
4. Fabric projector

The method of unraveling the cloth is used principally on dense fabrics with a felted surface where the separate yarns are hard to distinguish. This method is also sometimes used where, in addition to yarn count, the relative weights of warp and filling are being determined.

The pick glass is a small magnifying glass in a stand over a square exactly 1 inch each way (or sometimes ½ inch each way), so that if all the yarns in the field are counted the number of yarns per inch is directly determined. This is the method generally used.

The mechanical pick counter, such as the Suter, consists of a small microscope arranged to travel horizontally on a calibrated track. In the field of view is a needle-point. The microscope is placed on the cloth with the pointer at zero and is made to traverse the cloth, counting the yarns as they pass under the needle-point for a known distance. Then the number of yarns per inch is calculated.

The projector is simply a slide projector in which the cloth is placed between two plates with an opening of known dimensions. The number of yarns may be counted easily on the screen. This device is useful principally to avoid eyestrain. The *Readex* apparatus (originally designed for viewing microfilm) may also be used to project a magnified image of a textile fabric. This is useful not only for counting yarns but also for viewing defects.

If the pick glass is used, it is convenient to use a light table, a box with a ground glass top and containing several electric bulbs. The A.S.T.M. specifies that the number of ends per inch and picks per inch shall be counted in five different places, not counting the same threads twice and, of course, not near the selvedge. If there are fewer than twenty-five threads per inch, the number of threads in 3 inches shall be counted each time and the result divided by three. If the fabric is less than 3 inches wide, the total number of ends shall be counted and divided by the width.

In the case of pile fabrics, another pair of counts is of importance. The number of tufts or loops per inch, counted in the warpwise direction, is known as the *rows*; the number of tufts or loops per inch, counted in the fillingwise direction, is known as the *pitch*.

If the pattern of the cloth is very complicated, the number of yarns per inch has little meaning and the number of yarns to a pattern should be determined. This is really a job for a designer.

The construction of a cloth is usually designated by a combination of figures such as 90 X 56. This means ninety ends per inch and fifty-six picks per inch.

Weight

Two different weights are in use and may be determined in evaluating cloth: weight per unit area (ounces per square yard) and weight per unit length (ounces per running yard). Obviously, if either one and the width of the cloth is known, the other may be calculated (assuming the selvedge to have a negligible effect) by the formula:

$$S = \frac{36R}{B}$$

and

$$R = \frac{SB}{36}$$

where

S = weight per unit area (ounces per square yard)
R = weight per unit length (ounces per running yard)
B = width of cloth in inches

Another value often desired is the *yards per pound*. This is obviously equal to $\frac{16}{R}$ or $\frac{576}{SB}$.

Both weight determinations are based on cutting out pieces of cloth of known area and weighing. The value of *S* may then be calculated from the formulae:

$$S = \frac{36 \times 36}{A} \times \frac{G}{437.5} = 2.962 \frac{G}{A}$$

or

$$S = \frac{36 \times 36}{A} \times \frac{G'}{28.35} = 45.71 \frac{G'}{A}$$

where

A = area of sample in square inches
G = weight of sample at standard regain in grains
G′ = weight of sample at standard regain in grams

There are three general methods in use for determining the weight:

1. Weighing a large number of small areas, e.g., five pieces, each 2 inches square
2. Weighing one fairly large area, e.g., a piece 5 × 4 inches
3. Weighing a piece the full width of the cloth and 1 yard long. This gives the value of *R* directly.

In any of the above methods enough sample must be taken to give at least 20 square inches. The weighing should always be done when the sample is oven-dry and the results calculated to standard regain.

The cutting-out of the samples may be done by marking off the area with a ruler and cutting this area out with shears. However, if many determinations are to be made, it is better to use steel or glass templates, cutting around the templates with a sharp razor blade. Where a large number of small samples are to be cut out, steel dies may be used.

Many cloths are bought and sold by a description which includes the weight per running yard. This is possible only when a standard width is agreed upon. Woolen and worsted cloths are based upon a width of 54 inches, so that a sixteen-ounce worsted fabric is one of which a piece 54 inches wide and 1 yard long, would weigh 16 ounces at standard regain. Similarly, cotton duck is based on a width of 22 inches, and a sixteen-ounce duck would be one of which a piece, 22 inches wide and 1 yard long, would weigh 16 ounces at standard regain. Ducks are also given a number which is equal to 19 − R so that the duck mentioned above is 19 − 16. This is a No. 3 duck. A nineteen-ounce duck is No. 0 or 1/0; a twenty-ounce duck is No. 00 or 2/0; a twenty-one-ounce duck is 3/0, and so on.

The following examples will make clear the use of the formulae:

A piece of cotton cloth 4 × 5 inches weighs 1.55 grams when oven-dry. What is the weight per unit area?

$$G' = 1.55 \times 1.065 = 1.65 \text{ grams}$$

$$A = 4 \times 5 = 20 \text{ square inches}$$

$$S = \frac{45.71 \times 1.65}{20} = 3.77 \text{ ounces per square yard}$$

A sample of woolen cloth is to be tested for weight. Five pieces each 2 × 2 inches are cut out and weighed together; when oven-dry, they weigh 112 grains.

$$A = 5 \times 2 \times 2 = 20 \text{ square inches}$$

$$G = 112 \times 1.13 = 126.7 \text{ grains}$$

$$S = \frac{2.962 \times 126.7}{20} = 18.7 \text{ ounces per square yard}$$

$$R = \frac{18.7 \times 54}{36} = 28.1 \text{ ounces per running yard of cloth 54 inches wide}$$

A sample of duck is 21 inches wide and a length of 12 inches weighs, when oven-dry, 144 grams. What is the number of the duck?

$$A = 21 \times 12 = 252 \text{ square inches}$$

$$G' = 144 \times 1.065 = 153.3 \text{ grams}$$

$$S = 45.71 \times \frac{153.3}{252} = 27.8 \text{ ounces per square yard}$$

$$R = 27.8 \times \tfrac{22}{36} = 17 \text{ ounces per running yard; } 19 - 17 = \text{No. 2 duck}$$

Crimp

Crimp may be defined as the deviation from straightness of yarns as they lie in the cloth, expressed as percentage of length in the cloth. Take-up is the deviation from straightness of the yarns as they lie in the cloth, expressed as percentage of the original length of the yarn before weaving. The interrelationships of crimp and take-up may be shown by the following formulae:

L = length of yarn before weaving
L' = length of yarn in the cloth
C = crimp in per cent
T = take-up in per cent

then

$$C = \frac{L - L'}{L'} \times 100$$

$$T = \frac{L - L'}{L} \times 100$$

$$T = \frac{100C}{C + 100}$$

$$C = \frac{100T}{100 - T}$$

Since the process is sometimes reversed and it is desirable to know the original length of the yarn, from the above formulae we obtain:

$$L = \frac{L'(C + 100)}{100}$$

$$L = \frac{100L'}{100 - T}$$

In a fabric just off the loom, the warp threads generally have the most crimp because the filling yarns are drawn straight through the shed and the warp yarns are folded over and under the filling yarns by the up-and-down motion of the harnesses. Finishing operations such as stretching, calendering, and tentering may change the crimp.

A fabric which is to have all or most of the stress applied in one direction, such as duck belting, should have the minimum crimp in that direction, but fabrics such as balloon cloth, which are subject to equal stress in all directions, should have the same crimp in both warp and filling. This type of fabric is known as a *square* fabric. In general, the difference between warp and filling crimp is known as *off-square*. For example, if the warp crimp is 6% and the filling crimp is 4%, the cloth is 2% off-square.

It will be noted that crimp and take-up are the same quantity referred to different bases. The crimp is based on the length in the cloth and is of interest to the tester and the finisher; the take-up is based on the length before weaving and is of interest to the weaver and yarn manufacturer.

The principle of the crimp determination is very simple. With a fine pen and a rule, lines are drawn on a piece of cloth L′ distance apart. Some of the yarns are raveled out, the yarns are straightened without stretching, and the distance between the ink marks (L) is noted. The crimp is then calculated from the formula. The difficulty lies in the straightening of the yarn without stretching it. To do this, three methods are available:

1. Straighten by hand. This is inaccurate since we do not know the force applied.

2. Straighten by a standard weight. This is satisfactory if we know what weight to use. The determination of this weight will be given later.

3. Determine the straight length from the load-elongation curve. This is the most accurate method and the one which we shall take up in detail.

The usual method of crimp determination by load-elongation data is as follows: Make parallel ink lines on a piece of cloth L' distance apart; unravel five yarns and test one at a time. Clamp the yarn at one of the ink spots, allowing the yarn to hang vertically in front of a scale. Hang a small clamp of known weight on the yarn at the other ink spot. Read the elongation at this load. Apply successively small loads, reading the elongation at each new load until it is obvious that each additional load gives the same additional elongation. Plot the elongation horizontally and the load vertically to obtain a curve similar to Fig. 14.

In this curve, there is a curved portion (OB) and a straight-line portion (BC). The region OB represents the removal of the crimp. The region BC represents the stretch of the yarn. If there were no crimp, the curve would be all the straight line DC. Therefore, the distance OD represents the elongation of the yarn due to the removal of the crimp. Then the original length L′ plus the value OD is the length of the yarn before weaving (L) or

$$L = L' + OD$$

This method was originally devised by the Bureau of Standards. The yarn was loaded by attaching a light-weight scale pan

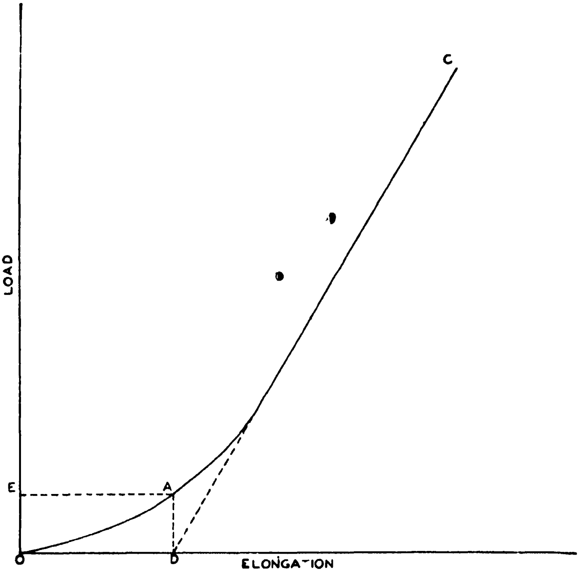

FIG. 14. Load-Elongation Curve for Crimp Determination

and adding weights. This method has been improved by using the chainomatic principle, loading the yarn by lowering a chain so that an increasing number of links of the chain are held by the yarn.

Saxl has devised a crimp determination apparatus in which the loading is done by a triple-beam balance and the elongation is read on a calibrated vertical rod. Recently, also, an automatic load-elongation device has been invented. This apparatus draws its own curve from which crimp may be readily obtained.

A simple and inexpensive apparatus may be made as follows: On a vertical board, fasten graph paper marked off in inches and tenths and fasten a small clamp at the top to hold the yarn. Obtain a small paper clamp with jaws $\frac{1}{4}$ inch wide and with a hook attached, such as is sold for use in schools to hang papers on a piece of cloth; also obtain a number of paper clips. Weigh the small clamp and some of the paper clips. Hang the yarn in the clamps and read the extension; add the paper clips, one at a time, reading the extension. A typical determination is given:

Weight of small clamp = 0.35 grams
Weight of 5 paper clips = 3.90 grams
Weight per paper clip = 0.78 grams

Weights Added	*Elongation Inches*	*Load Grams*
0	0	0
clamp	0.21	0.35
clamp + 1 clip	0.37	1.13
clamp + 2 clips	0.41	1.91
clamp + 3 clips	0.45	2.69
clamp + 4 clips	0.49	3.47
clamp + 5 clips	0.53	4.25

On plotting the above and extending the straight line portion, it was found that OD = 0.31 inches. Therefore,

$$L' = 10 \text{ inches} \qquad L = 10.31 \text{ inches}$$

$$C = \frac{0.31}{10} \times 100 = 3.1\% \text{ crimp}$$

It is not always necessary to plot the data, provided that equal intervals of load are taken. Examination of the data will show when a straight line is obtained. Then, if any two points on the straight line are taken

$$OD = \frac{E_1W_2 - E_2W_1}{W_2 - W_1}$$

where

E = elongation in inches
W = load in any units

If the crimp is to be determined by the length with a standard weight applied, it is necessary to have one weight for each type of yarn and fabric. This is satisfactory in a mill where only one class or a few classes of goods are being made. The standard weight to be used is determined from the load-elongation curve by projecting from D to the curve and from the curve over to the load axis. In Figure 14 the distance OE represents the weight which would be just sufficient to remove the crimp. Another way of determining the standard weight is to use the A.S.T.M. formula:

$$OE = \frac{131}{\text{Typp number}}$$

This formula is merely an approximation and is not as accurate or desirable as the previous method.

It has been found that temperature and relative humidity have only a very slight effect on the crimp, so that standard conditions are not necessary for crimp testing.

Crimp may also be determined from the strength-stretch curve which will be taken up later, but the results are not as accurate as the methods just described due to the small scale of the curve.

A typical problem involving crimp is as follows: A sample of cotton cloth, 6 inches square, is picked apart and yields 360 warp yarns, which weigh 0.955 grams when oven-dry, and 320 filling yarns, which weigh 1.150 grams when oven-dry. A crimp determination gave 4.2% crimp in the warp and 3.6% crimp in the filling. What is the cloth count, the weight of the cloth, and the counts of the warp and filling?

Cloth Count:

$$\text{Warp } \tfrac{360}{6} = 60 \text{ ends per inch}$$
$$\text{Filling } \tfrac{320}{6} = 53.3 \text{ picks per inch}$$

Weight:

$$\begin{array}{rl} \text{Warp} = & 0.955 \text{ grams} \\ \text{Filling} = & \underline{1.150} \text{ grams} \\ \text{Total} \quad & 2.105 \text{ grams} \end{array}$$

$$2.105 \times 15.43 \times 1.065 = 34.6 \text{ grains at standard conditions}$$

$$S = 2.962 \frac{G}{A} = 2.962 \times 34.6/36 = 2.84 \text{ ounces per square yard}$$

Counts:

Warp

$$L' = 360 \times 6 = 2160 \text{ inches} = 60 \text{ yards}$$

$$L = \frac{L'(100 + C)}{100} = \frac{60 \times 104.2}{100} = 62.6 \text{ yards}$$

$$W = 0.955 \times 15.43 \times 1.075 = 15.8 \text{ grains}$$

$$N = \frac{L}{W} \times \frac{7000}{H} = \frac{62.6}{15.8} \times \frac{7000}{840} = 33.0\text{'s}$$

Filling

$$L' = 320 \times 6 = 1920 \text{ inches} = 53.3 \text{ yards}$$

$$L = \frac{53.3 \times 103.6}{100} = 55.2 \text{ yards}$$

$$W = 1.150 \times 15.43 \times 1.075 = 19.0 \text{ grains}$$

$$N = \frac{55.2}{19.0} \times \frac{7000}{840} = 24.2\text{'s}$$

Thickness

The determination of the thickness of a textile material is a delicate operation because of the ease of compression of most fabrics. For this reason, thickness is seldom determined except upon felts, ducks, or other fairly rigid fabrics. If the thickness of a soft material must be determined, it is better to do it microscopically on a cross-section of the fabric. This is especially true of pile fabrics.

The usual type of thickness gage (Fig. 15) has a broad anvil upon which a presser foot is pressed by a spring. The sample is placed on the anvil and the presser foot is lowered onto the sample by releasing the raising lever very slowly, the dial then indicates the number of mils or thousandths of an inch between the anvil and the presser foot.

Haven has listed the variables which affect the result and has attempted to standardize these variables in the following manner:

1. Size of presser foot. The greater the size of the presser foot the less it will sink into the fabric. One inch is suggested.

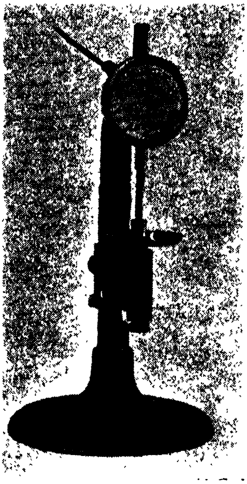

FIG. 15. Thickness Gage

2. Form of presser foot. Haven suggests circular for cloth and square for cords.

3. Weight on presser foot. The greater the weight, the deeper the presser foot will sink into the cloth. Two ounces are suggested.

4. Velocity of presser foot. The faster the foot is allowed to drop, the more it will penetrate the fabric. Haven suggests a clock-work mechanism which would lower the presser foot at the rate of 12 inches per minute.

5. Time elapsing during measurement. The presser foot does not come to equilibrium immediately. Five seconds are suggested.

6. Stability of the machine. Jarring will cause the foot to penetrate more deeply. A solid support is necessary.

7. Condition of the specimen during measurement. The fabric should be free of wrinkles but not under tension.

The A.S.T.M. has standardized the following method of using the thickness gage: The presser foot shall be circular and 0.375 ± 0.001 inches; the presser foot and anvil shall be plane and parallel to within 0.0001 inches; the total weight on the presser foot shall be 6.0 ± 0.1 ounces (3.4 pounds per square inch). The fabric shall be placed on the anvil smooth and without tension; the presser foot shall be lowered gradually (without impact) and allowed to rest ten seconds before the dial is read. Not less than five measurements shall be made on a sample and the results averaged.

The A.S.T.M. also modifies the above specifications in the case of pile fabrics and wool felts as follows:

Total thickness of pile floor coverings
  Pressure = 0.100 ± 0.001 pounds per square inch
  Diameter of foot = 3.00 ± 0.01 inches
Thickness of backing of pile floor coverings
  Pressure = 0.750 ± 0.001 pounds per square inch
  Diameter of foot = 1.00 ± 0.01 inches
Thickness of wool felt
  Pressure = 10 ± 0.5 ounces
  Diameter of foot = 1.129 inches

In spite of all these precautions, however, the values obtained are of questionable accuracy, although they do have some value in the comparison of different samples done on the same apparatus, by the same person, and using the same technique.

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Chapter 6

FABRIC TESTING—PROPERTIES DEPENDENT UPON AIR SPACE

The number and size of the air spaces in a fabric greatly affect certain properties of the fabric. Even if the total amount of air space in two fabrics is the same, it is obvious that the one with a large number of small spaces will allow less air to pass and will be warmer than the one with a smaller number of large spaces. Porosity is the amount of air space in a fabric and permeability is the ability of the fabric to allow a gas or vapor to pass through it. The thermal properties of the fabric are, as a rule dependent upon the amount of air space as well as the size of the spaces since, in general, the solid fibers themselves are not good heat insulators. However, air spaces trapped by the fibers may be very efficient insulators, if the air is not in motion.

Porosity

Porosity is determined by measuring the total volume of a fabric and calculating the total volume of fiber in the sample. The difference between these two values is air space and, when calculated as per cent of the total volume, gives the porosity. If we let

S = total volume of sample in cubic centimeters
F = total volume of fiber in cubic centimeters
A = area of the sample in square centimeters
W = weight of the sample at standard regain in grams
T = thickness of sample in centimeters
D = density of fiber in grams per cubic centimeter (Table X)
P = porosity in per cent

By definition,

$$P = \frac{100(S - F)}{S}$$

also,

$$S = AT \text{ and } F = \frac{W}{D}$$

then,

$$P = \frac{100\left(AT - \frac{W}{D}\right)}{AT}$$

TABLE X

DENSITIES OF DRY FIBERS

	Grams per Cubic Centimeter
Nylon	1.14
Elastic vinyl fiber	1.19
Silk (boiled-off)	1.25
Tussah	1.27
Casein fiber	1.29
Wool, mohair, camel hair	1.32
Acetate rayon	1.33
Vinyl fiber (regular)	1.35
Jute, hemp	1.48
Cotton, linen	1.50
Ramie	1.51
Viscose, cupra, saponified acetate rayons	1.52
Vinylidene fiber	1.72
Glass fiber	2.56
Asbestos	2.10–2.80

An example will show the calculations necessary. A cotton fabric, 6 inches square, gives an average thickness on the gage of 11.0 mils (0.0110 inches) and, when oven-dry weighs 3.65 grams. What is the porosity? The English units must first be converted to metric units (Table VIII):

$$6 \text{ inches} = 6 \times 2.54 = 15.2 \text{ centimeters}$$
$$0.0110 \text{ inches} = 0.0110 \times 2.54 = 0.0279 \text{ centimeters}$$

Then, using the formula

$$A = 15.2 \times 15.2 = 231 \text{ square centimeters}$$
$$T = 0.0279 \text{ centimeters}$$
$$W = 3.65 \times 1.065 = 3.88 \text{ grams}$$
$$D = 1.50 \text{ (from Table X)}$$
$$P = \frac{100\left(231 \times 0.0279 - \frac{3.88}{1.50}\right)}{231 \times 0.0279} = \frac{100(6.45 - 2.59)}{6.45}$$
$$P = 59.8\%$$

The volume of fiber (F), instead of being calculated from the weight, may be measured directly by the Russell Volumeter. This consists of a bulb and calibrated tube connected at the tube end by a ground glass joint to a bottle; the bottle and bulb have approximately the same volume. Enough carbon tetrachloride is placed in the bulb to bring the level into the calibrated part of the tube. The bottle is put in place and the whole apparatus is inverted. The volume is then read. The apparatus is reinverted, the fabric placed in the bottle, the bottle replaced, inverted to its reading position, and the new volume is read. The difference in the two readings is the volume of fiber. This apparatus can also be used to measure the density of fiber if a known weight of fiber is used.

Porosity is valuable in giving an idea as to the permeability, warmth, and lightness of a fabric. Although these qualities may be measured separately, they require special apparatus, whereas porosity requires only a thickness gage in addition to an oven and balance. Permeability is only roughly proportional to porosity, the type of finish on a cloth having considerable effect on the permeability. For example, a cloth, before and after felting, might have the same porosity but would be less permeable after felting. The warmth of a fabric is due to dead air spaces in the fabric, so the most porous fabric would be the warmest, provided that it is not, at the same time, too permeable, i.e., the air spaces are not too large. The lightness of the fabric is obviously proportional to the porosity since both qualities involve the weight of a given area.

Permeability

Two types of permeability are of interest:

1. Permeability to air. This is desirable in clothing but undesirable in overcoating, sailcloth, balloon cloth, airplane fabric.

2. Permeability to water vapor. This is desirable in clothing, especially in underclothing, but undesirable in materials to be used for coverings, such as tarpaulins.

PERMEABILITY TO AIR

Permeability to air may be measured by one of several methods:

1. By forcing air through a fabric at a constant rate and measuring the back pressure developed. Haven has described an apparatus for this purpose. The apparatus however, is rather complex and expensive.

2. By forcing air through a fabric at constant pressure and measuring the rate of flow of the air. The most frequently used apparatus for this process is the Frazier machine (Fig. 16). A suction fan, which can be regulated to various speeds, draws air through a known area of fabric which is clamped over a circular orifice. A manometer measures the pressure drop across the fabric and another manometer measures the pressure drop across the calibrated orifice; by means of this second manometer reading, the volume of air which is flowing may be measured. A sample, 10 inches square, is mounted between the clamp and orifice with sufficient tension to smooth the fabric. The fan is started and its speed regulated until the pressure drop across the fabric is a specified value (usually 0.5 inches of water). The orifice manometer is read and, from the calibration chart, the flow of air is recorded in cubic feet per minute per square foot of fabric at the stated pressure drop. Several orifices are supplied to take care of fabrics of different degrees of permeability.

3. By forcing a known volume of air at a standard pressure through a fabric and measuring the time required. This method

may be illustrated by the Gurley Densometer or by an apparatus which the author has devised.

The Densometer consists of a cylinder about 12 inches high with an orifice of definite diameter at the bottom, against which is pressed a companion orifice. The fabric is inserted between the two orifices. The cylinder is open at the top and contains an inverted cylinder slightly smaller in diameter than the outside cylinder. A tube runs from the top orifice nearly to the top of the cylinder. The annular space between the tube and the cylinder contains water or, better, an oil of known viscosity up to a definite level. In operation, the inside cylinder is raised, drawing in air, the fabric is placed between the two orifices and the inner cylinder is released, forcing the air out through the fabric. The time for the cylinder to fall between two rings marked upon it is noted. A stop watch or an automatic timing

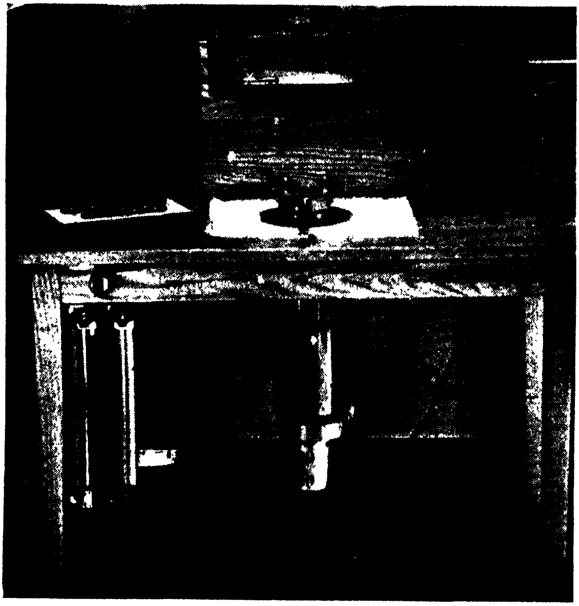

FIG. 16. Frazier Permeability Tester

attachment may be used. The greater the permeability of the fabric to air, the less time is required.

In the case of fabrics of low permeability, the Densometer is satisfactory, but fabrics of high permeability give a very short measured time on the Densometer and the results are, therefore, somewhat inaccurate. This may be offset by the use of a smaller orifice, but then the sampling error is apt to be excessive. It is safer not to use the Densometer on very loosely woven fabrics.

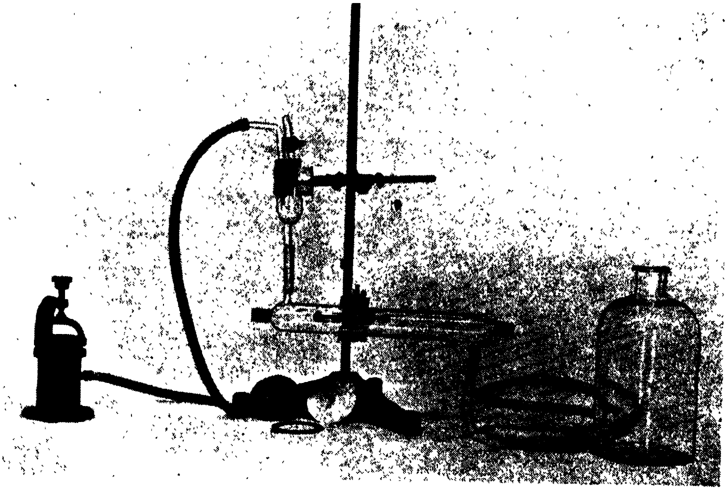

FIG. 17. Apermeter

The author's device, the Apermeter, is illustrated in Fig. 17. The essential parts are a condenser jacket the ends of which are sealed with rubber stoppers, a funnel and an aspirator bottle. There is also a sample holder which is the one used in the hydrostatic pressure tester for water resistance (see later). The apparatus is so arranged that, when the aspirator bottle rests on the table, the water level is about ½ inch below the bottom outlet of the condenser jacket. The funnel connected to the jacket has a rubber stopper containing a glass stopcock and an elbow, the elbow being connected by rubber tubing to the sample holder.

The sample is placed between two thin sheets of metal which have concentric three-eighth-inch holes. The stopcock is opened and the aspirator bottle is raised until the jacket and about half of the funnel are filled with water. A pinchcock is then placed on the tubing at the aspirator bottle, the bottle is placed on the table, and the stopcock is then closed. The pinchcock is released and the stop watch is started as the water level passes the entrance to the jacket and stopped when it passes the exit of the jacket. A run is made each day with no sample in place and the time (t_0) observed. Then

$$\text{Per cent permeability} = 100\,\frac{t_0}{t}$$

This apparatus is simple to build and to operate. The parts are standard laboratory apparatus and all variables, such as variations in pressure, sample area, and changes in position, are cancelled by the blank run with no fabric.

Permeability to air has been suggested as a method of measuring the progress of the fulling operation; commercially fulled fabrics are 22 to 59% less permeable to air than the same fabric when unfulled. The use of this determination, then, might make a given degree of fulling more readily reproducible.

PERMEABILITY TO WATER VAPOR

The permeability of a fabric to water vapor has been measured by three different methods:

1. By covering a vessel containing concentrated sulfuric acid with the fabric to be tested and exposing to a standard atmosphere for a definite length of time then measuring the increase in weight of the sulfuric acid. This method requires considerable standardization to give comparable results.

2. By covering a vessel containing water at 37.5° C. (body temperature) with the fabric to be tested and exposing to a standard atmosphere for a definite length of time then measuring the decrease in weight of the water. This requires even more standardization.

3. By covering a vessel containing water at any temperature with the fabric to be tested and exposing to any atmosphere for any convenient time, also exposing a similar vessel of water with no covering to the same atmosphere and for the same time. The permeability is then given as the ratio of the loss through fabric to the loss from open vessel. This method is much simpler than either of the others and is the one used by Black and Matthew in determining the permeability to water vapor of various underclothing materials.

Thermal Properties

The thermal property of a fabric is its ability to resist or permit the passage of heat. In winter clothing, of course, warmth is desired; in summer clothing, coolness is necessary. Intimately associated with thermal properties are porosity and permeability, since the warmth of any fabric is due to the dead air space and not actually to the fiber itself. Furthermore, to be comfortable, clothing must allow evaporation of moisture from the body and so must have a certain amount of permeability. We have already considered porosity and permeability and shall now concern ourselves with the measurement of thermal properties alone.

There are three common methods of measuring the thermal insulating value of fabrics:

1. Disc or plate method
2. Constant temperature method
3. Cooling method

DISC OR PLATE METHOD

In the disc or plate method, the fabric is held between two plates at different temperatures and the rate of flow of the heat through the fabric is measured. If pressure is exerted on a fabric, its properties are altered. If the pressure is great enough to get good thermal contact between the plates and the fabric, the change in properties is considerable. If the pressure is small, the thermal contact is poor and the surface resistance to the flow of heat may be large enough to cause serious error in the determination. Results obtained by this method, therefore, are the thermal conductivities of compressed fabrics under a pressure which must be stated, if the results are to be reproducible. Speakman's modification of this method consists in using two calorimeters with the fabric between supported at constant thickness.

The thermal conductivity of any material is given by the formula:

$$k = \frac{QL}{TA(t_1 - t_2)}$$

$$C = \frac{k}{L} = \frac{Q}{TA(t_1 - t_2)}$$

where

Q = heat transmitted in calories
L = thickness of material in centimeters
A = area of sample in square centimeters
T = time in seconds
$t_1 - t_2$ = temperature difference between the two sides of the material in degrees Centigrade
k = thermal conductivity per centimeter of thickness
C = conductance of the particular sample

It is obvious that this method involves accurate heat measurements as well as accurate thickness control.

The method using the Fitch thermal conductivity apparatus is perhaps the most commonly used for testing heat transfer properties of fabrics. The apparatus shown in Fig. 18 consists of a *source* which holds water maintained at the boiling point by an immersion heater, a *receiver* which consists of a known mass of copper insulated from its surroundings except at the top, and a micro-ammeter or galvanometer. The micro-ammeter is connected to two thermocouple junctions, one in the bottom of the source and one in the top of the receiver. To insure proper contact with the sample (placed between the source and the receiver), the source should have a weight on top so that the total weight of source, water, weight, and heater applied to the face area of the receiver will give a pressure of 3.4 pounds per square inch, the same pressure that is used in making thickness measurements.

This is best done by having a known weight on top, e.g., about 1900 to 2000 grams, and then regulating the final weight by the amount of water placed in the source. For example, in one particular case, the diameter of the receiver face is 1.75 inches or 4.45 centimeters; the area then is 2.405 square inches or 15.52 square centimeters. Since the standard pressure is 3.4 pounds per square inch, 3.4 × 2.405 or 8.19 pounds total weight are required; this is equal to 8.19 × 454 or 3720 grams. The weight of empty source is 909 grams, the heater is 152 grams, and the steel plate used as a weight is 1930 grams, mak-

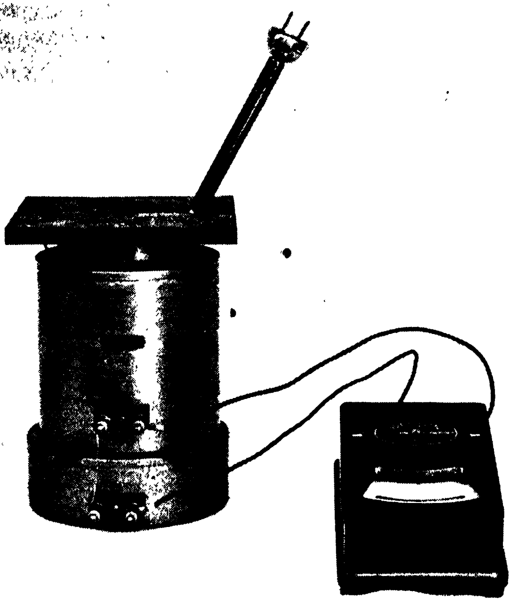

FIG. 18. Fitch Thermal-conductivity Apparatus

ing a total weight of 2990 grams. Then, 3720 − 2990 or 730 grams of water should be placed in the source.

The material to be tested is placed below the source, but not on the receiver, and the source is heated to boiling. The source and sample are then lowered on the receiver, and the current is read every 1, 2, or 3 minutes, according to the rate of change. About ten readings should be taken altogether. Record the time (t), current (I), and logarithm of the current. A plot of log I against t should be made and the slope (S) of the resulting straight line obtained. It may be shown mathematically that

$$t = -\frac{2.303LMc}{kA}(\log I - \log I_0)$$

where

M = mass of copper in grams (stamped on apparatus)
c = specific heat of copper = 0.093 calories per gram
A = area of receiver face in square centimeters

Then,

$$S = \frac{2.303LMc}{kA}$$

or

$$\frac{k}{L} = \frac{SA}{2.303Mc} = C$$

This is the conductance of the particular fabric tested. If the coefficient k is desired, the thickness L must also be measured and

$$k = CL$$

CONSTANT-TEMPERATURE METHOD

In the constant-temperature method, a body is wrapped with the fabric to be tested and is maintained at a constant temperature by a controlled source of energy (usually an electric heater). The amount of energy required to maintain the body at the fixed temperature is determined. In this way, an electrical measurement, which is easier and more accurate than the measurement of heat energy, is made. This method has been used by Haven, the Bureau of Standards, Marsh, and Angus in making very accurate comparisons of thermal insulating values. Absolute determinations cannot be made. Therefore, comparisons of results are possible only when the same instrument is used.

COOLING METHOD

In the cooling method, we have a hot body wrapped in the fabric to be tested and the rate of cooling is measured or, more usually, the time required for the body to cool a given number of degrees. The results apply only to the apparatus used, i.e., the results may not check when two different instruments are used, but for comparisons of different fabrics in one laboratory the simplicity of the method and apparatus makes it very valuable. Black and Matthew have used this method using a katathermometer, i.e., an alcohol thermometer with a cylindrical bulb about 4 centimeters long, 2 centimeters in diameter, and a stem 20 centimeters in length with two graduations at 100° F. and 95° F. (temperatures just above and just below body temperature). It was found that relative humidity had no effect on this apparatus. The cloth is made into a cylinder by being wrapped on a wooden form, marked, and sewed. Tabs are left at the bottom and top of the cylinder to fasten on ebonite rings by means of shellac. The whole apparatus is placed in an enclosure at constant temperature (18.5° C. was used by Black and Matthew) and the time of cooling from 100° to 95° F. is noted with the fabric and without the fabric. The thermal insulating value is given as the ratio of time of cooling unclad divided by time of cooling when clad. This ratio varies from 0.69 to 0.96 for the *comfort zone.*

The use of these different methods of measuring thermal insulating value has shown several very important facts about the warmth of fabrics. The fiber used is of importance in the warmth of the fabric only as it affects the porosity and permeability of the fabric or the permanence of the warmth. For example, cotton and woolen fabrics may be made identical in warmth but after use the cotton would mat down and be less porous, i.e., have less dead air space, whereas the resilience of the wool fabric would bring it back to its original porosity. The cotton fabric would, therefore, be less warm after use, whereas the wool fabric would retain its warmth. It has also been shown that napped fabrics are warmer than unnapped and loose garments are warmer than tight. These effects are due to the dead air space formed. Fulling decreases the permeability to air but increases the warmth of a woolen fabric; the porosity remains about the same; fulling has decreased the size of the individual air spaces but increased the number of such spaces so that a greater warmth is obtained.

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Chapter 7

FABRIC TESTING—WATER AND FABRIC RELATIONSHIPS

Absorbability and Wetting-out

Absorbability and wetting-out both refer to the entrance of liquid into a yarn or fabric. Absorbability is the ability of the fabric to take up a liquid. Wetting-out is a technical term meaning the ability of a liquid to enter a fabric and displace the air from the capillary spaces. Both terms refer to the same phenomenon but absorbability refers to the fabric and wetting-out to the liquid. Two factors are important in absorbency: the total amount of liquid absorbed and the rate of absorption of the liquid. Sometimes one factor is measured and sometimes the other.

Absorbability is also related to the warmth of a fabric. If a fabric is permeable to air but does not absorb water, evaporation of perspiration takes place from the skin and the skin temperature falls. This is the phenomenon which occurs when nylon fabrics are worn. If the fabric absorbs the perspiration, however, the evaporation takes place from the fabric and not from the skin; therefore, chilling does not occur.

The methods used in testing fabrics for absorbency and testing wetting-out agents (or penetrants) are:

1. Surface tension. This does not take capillary action into account and so is not always comparable with practical results. For the determination of surface tension see any text on physical chemistry or colloid chemistry.
2. Sinking time of a tuft of fiber or yarn or a patch of cloth.
3. Draves' method. This is a standard method of the A.A.T.C.C.

4. Capillary travel method. The rate of absorption is measured in this method.

   a. Wick-up or modified Weireck method
   b. Haven's method

5. Absorption from a wet solid surface such as a wet brick or tile. The total absorption is measured in this method.
6. Absorption on immersion in a liquid. The total absorption is measured in this method.
7. Absorption on spraying with water. This is a standard method of the A.S.T.M. The rate and amount of absorption are measured in this method.

SINKING TIME

The sinking time of a patch of cloth is a test very often carried out in the industry because it is simple to do, requires no special apparatus, and is quick. The results obtained by one operator, however, are never comparable with those obtained by another operator or even with results by the same operator at different times. The usual method of testing is to place the liquid in a series of beakers. The fabric is then cut into a number of squares of equal size ($1\frac{1}{2} \times 1\frac{1}{2}$ inches). The test consists of dropping a square on the surface of the liquid (preferably from a standard height) and measuring with a stop watch the time required for the square to sink. The watch is started when the fabric strikes the surface and is stopped when the last corner sinks below the surface.

DRAVES' METHOD

Draves' method which is a standard method of the A.A.T.C.C., is an elaboration and improvement on the method above (see Fig. 19). It measures the time required for a standard skein to sink in a solution of wetting agent when the skein, carrying a standard weight, is held below the surface of the wetting solution by an anchor. The sinker consists of No. 10 copper wire, $2\frac{1}{2}$ inches in length, bent into the form of a hook. The weight of the sinker should be 3.00 grams. It is also desirable to use hooks of 1.50 grams and 6.00 grams in order to obtain a complete description of the wetting properties.

The sinker is fastened by a fine, strong thread at a distance of 1 inch from an anchor which should weigh at least 40 grams. A five-gram skein of gray, two-ply cotton yarn is folded once to form a loop, the sinker and anchor are attached, and the opposite end of the skein is cut through with shears. The wetting solution in a graduated cylinder with a capacity of 500 cubic centimeters is brought to 25° C., 50° C., 70° C., or 90° C., according to the conditions under which the wetting agent is to be used, and the skein is dropped in.

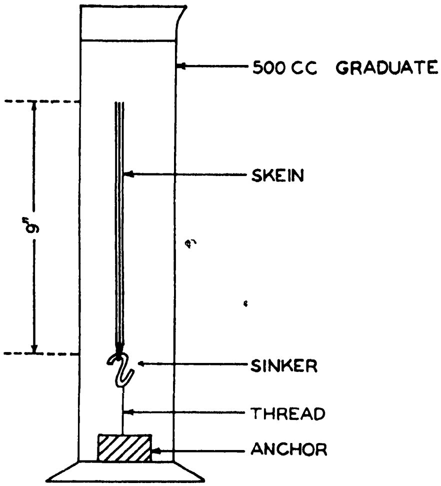

FIG. 19. Draves Wetting-out Test

The time elapsing between the entrance of the skein and the moment when the weighted skein starts to sink (when the thread between the sinker and anchor slackens) is measured with a stop watch. The average of at least four tests should be taken. Wetting agents are tested in concentrations of 1, 2, 5, 7, 10, 20 grams per liter and the results plotted. Different wetting agents are compared by determining the concentrations which will give a sinking time of 25 seconds. If this method is to be used for testing yarns or fabrics for wetting-out, a 1½ gram sinker or even a ½ gram sinker may be used, since the three-gram sinker may be too heavy for samples which wet-out readily.

CAPILLARY TRAVEL METHODS

Wick-up Method

The wick-up method for testing towelling uses strips of fabric, 6 inches long and ⅞ inch wide, cut both warpwise and fillingwise. A 0.005% water solution of Pontacyl Rubine R Conc. (Colour Index No. 179) is used so that the water level may be seen more readily. Each strip is suspended alongside of a ruler, with 1 inch immersed in the liquid, a hook and a two-gram weight keeping the sample straight. The heights to which the liquid rises in 15, 60, 90, and 120 seconds are observed and the results plotted. As a routine test, the two-minute test only may be used.

Haven's Method

Haven's method also measures the rapidity of absorption. Strips of cloth, 1 inch wide, are cut from selvedge to selvedge. These strips are held horizontally over the ground-glass top of a light box with a scale alongside the edge of the ground glass. The tension in the strips should be made uniform by attaching standard weights at one end of the strips. The weighted end is allowed to dip into a tray of water, and the position of the water in the strips is read at various time intervals. A plot of capillary travel against time is prepared and used in rating the fabrics for absorbency. Hess and Reidheimer have shown that to get reproducible results a constant relative humidity and temperature are necessary in the Haven method.

ABSORPTION FROM A WET SURFACE

Larose's method for water absorption from a porous plate is the most satisfactory test of this type. A porous plate is immersed in water which is under a slight head of water pressure so as to keep it moist. The sample is placed in contact with the plate, a glass slide and weight are put on top, and the sample is allowed to remain for varying periods of time. The per cent increase in weight of the sample is calculated.

ABSORPTION ON IMMERSION

The A.A.T.C.C. immersion test is shown in Fig. 20. Any tank, which gives an eight-inch depth of water, may be used.

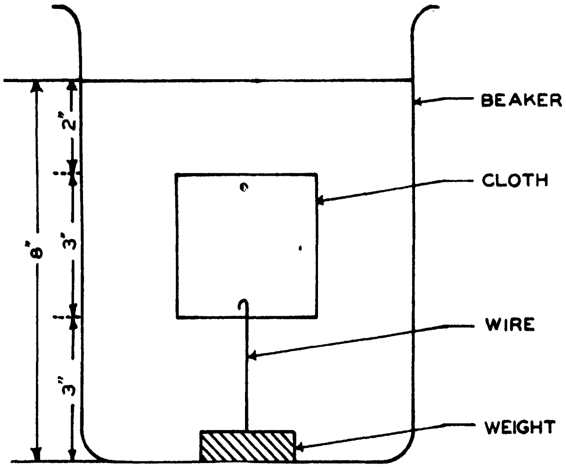

FIG. 20. Immersion Test

A sample, 3 × 3 inches, is conditioned and weighed. A sinker and hook are inserted in the middle of one edge, and the whole is dropped into the water which should be at 80° F. ± 2°. After 20 minutes, the sample is removed, placed between two sheets of standard blotting paper* 7 × 8 inches, and passed through a

* The blotters should meet the following specifications:
Weight—19 × 24 inches, 500 — 120 pounds
Caliper—26 to 32 points
Absorbency—2 to 6 seconds TAPPI Official Standard T-432 m-42 (Water absorption of Bibulous Paper)
Blotting papers, which meet these specifications, have been obtained

wringer at a speed of 1 inch per second. The sample is then reweighed, and the per cent increase in weight is calculated.

ABSORPTION ON SPRAYING

The A.S.T.M. absorbability test measures the absorption on spraying. The spray nozzle is 2¼ inches in diameter and contains eighteen holes evenly spaced but with no hole in the exact center. By means of a constant head of water, the spray is adjusted to allow 500 cubic centimeters per minute to flow. The spray falls 24 inches on the fabric (held between two concentric hoops, each 6 inches in diameter) for 1 minute. The fabric is then removed and placed between sheets of filter paper. It is rolled with a thousand-gram roller. A four-inch square is then cut out and weighed. This square is dried in the oven and weighed again. The per cent water absorbed is reported as per cent of the dry weight.

In testing wetting-out agents or penetrants, the rate of wetting is all that is required; but in testing a fabric for absorbency, both rate of absorption and total absorption should be determined since a towelling, for example, might absorb water very rapidly but not hold very much or, on the other hand, might absorb a large amount of water but take it up too slowly to be of value.

Water Resistance

Before considering methods of measuring water resistance, we must note the ways in which water can pass through a fabric. These ways are:

1. By wetting the fabric, followed by capillary action which brings the water to the other side.

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under the following designations from the sources listed:

19 × 24—120 Buff Albemarle Verigood Blotting
Albemarle Paper Mfg. Co., Richmond, Va.
19 × 24—120 Buff Sterling Blotting
Standard Paper Mfg. Co., Richmond, Va.
19 × 24—120 Parker's Buff Commercial Blotting
Joseph Parker & Sons, New Haven, Conn.

2. By the pressure of the water, forcing it through the openings of the fabric.
3. By a combination of the two actions already mentioned.

If a fabric were made in which there were no openings between the yarns, the cloth might still allow water to pass, if the water wet the fibers. This is what happens in closely woven duck or canvas. If a fabric of ordinary weave were made of fibers which had been chemically treated so that they would not be wet by water, the cloth would allow much of the water to roll off without penetrating, but if the water gathered in a thick layer on the cloth or if the water struck the cloth with much force, it would pass through the openings. This is the case in the so-called *showerproof* fabrics.

The only way to completely waterproof fabrics is to fill the openings and coat the fabrics with substances which are not themselves penetrated by water. Examples of such substances are: rubber in ordinary raincoat's, boiled oils in oilskins, tars or bitumens in tarpaulins, waxes in some tent cloths or plastic-coated fabrics. Fabrics treated with such substances are also impermeable to air and, therefore, are unsuitable for wear except in extreme cases. For ordinary wear, only a moderate degree of water resistance, combined with permeability to air and water vapor, is necessary. Strictly speaking, then, we are not measuring water-proofness but water resistance or water repellence.

The following methods are now used for measuring water resistance. Each method has been used in many variations, but only one variation of each will be given.

1. Drop penetration test (A.S.T.M.)
2. Spray test
   a. Ordinary spray (A.A.T.C.C.)
   b. Impact spray (A.A.T.C.C.)
3. Hydrostatic pressure test (A.S.T.M. and A.A.T.C.C.)
4. Contact angle or wetting angle (no standard test)

DROP PENETRATION TEST

The A.S.T.M. drop method is the most common form of drop penetration test. An overflow reservoir supplies water at constant head to a stalagmometer which is adjusted to give drops weighing 0.100 ± 0.003 grams at the rate of ten drops in 7 seconds. The sample is mounted 30 ± 0.5 centimeters below the tip of the stalagmometer and fixed at an angle of 45°. It is placed on a square plate of rubber or other nonconducting material upon the surface of which are two metal strips exactly 1 centimeter apart. These metal strips are part of an electrical circuit which contains a lamp. A piece of filter paper, which has previously been saturated with a 5% solution of sodium chloride or potassium nitrate and dried, is placed across the strips. The four-inch square sample is placed on top, and drops are allowed to fall until the lamp lights up. The time is measured with a stop watch.

SPRAY TEST

The A.A.T.C.C. spray method is illustrated in Fig. 21. The cloth is held between two hoops at an angle of 45° to the spray. The spray nozzle is a standard one. The distance from the bottom of the spray to the center of the fabric is 6 inches. Two hundred and fifty cubic centimeters of water, at 80°F. ± 2°, are poured into the funnel and allowed to spray the fabric. The fabric and hoops are then tapped on a solid object with the fabric down, rotated 180° and tapped again, and the fabric is matched against the rating chart (Fig. 21) for evaluation. Interpolations cannot be made.

IMPACT SPRAY TEST

The A.A.T.C.C. impact penetration test (Fig. 22) is also a spray test but made under more severe conditions. The nozzle is a different type and is located 24 inches above the center of the sample. A previously weighed standard paper blotter, 6 × 9 inches, is placed below a 7 × 13 inch sample of fabric

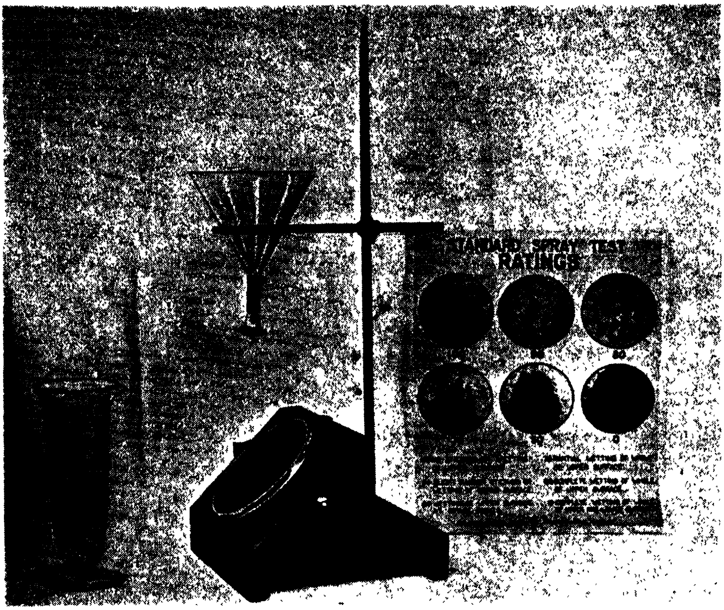

FIG. 21. Spray Test and Rating Chart

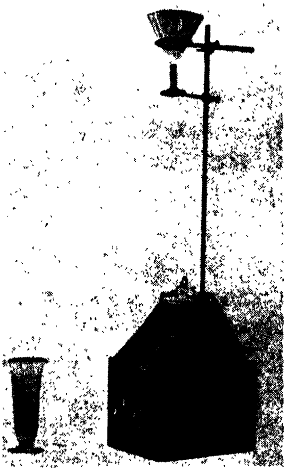

FIG. 22. Impact Spray Test

on the holder and a spring clamp holds the sample taut. Five hundred cubic centimeters of water at 80°F. ± 2° are poured into the funnel and allowed to spray the sample. The test specimen is then lifted; the blotter is removed and weighed again. The increase in weight of the blotter is reported to the nearest 0.1 gram.

HYDROSTATIC-PRESSURE TEST

There are several hydrostatic-pressure testers available. The A.A.T.C.C. apparatus (Fig. 23) is the most frequently used.

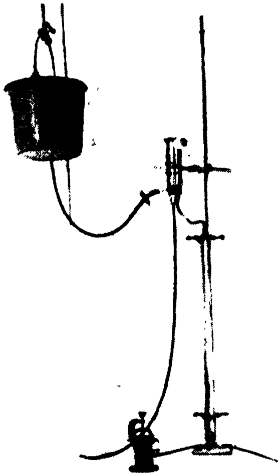

FIG. 23. Hydrostatic-pressure Test

The sample is clamped on a well by a clamp. The well is connected by rubber tubing to a glass tube, behind which is a scale indicating up to at least 50 centimeters. An overflow constant head device delivers water at 80° F. ± 2° to the top of the tube at such a rate that the level in the tube rises 1 centimeter per second. In use, the well is filled with water, and the scale height is adjusted to zero. The sample is clamped in place, and water is run into the pressure tube until three drops of water appear on the upper surface of the fabric. The flow of water is immediately stopped, and the height of water is reported. No definite standards are set up, but it is generally believed that a pressure of 17 centimeters or more indicates a fabric is showerproof; a pressure of 50 centimeters indicates that it is rainproof; and if it stands 50 centimeters for 1 hour, it is waterproof.

The A.S.T.M. has two similar hydrostatic-pressure tests in which the fabric forms the bottom of a cylinder. This apparatus is less convenient than the A.A.T.C.C. type and is not used so frequently. The Suter hydrostatic tester is still in use but is no longer a standard method.

CONTACT-ANGLE TEST

Several methods of measuring the contact angle between water and fabric have been developed for use in research. However, these are rather slow and unsuitable for control purposes, although in theory, they are most suitable for measuring water resistance.

EFFECT OF LAUNDERING AND USE

In evaluating the water resistance of fabrics, the effects of laundering or dry cleaning and use should also be considered. Smith has devised an apparatus which accelerates aging of the fabric and enables one to predict how the cloth will behave after use.

Experience in the field, as well as numerous service tests by the U. S. Army, has led to some facts regarding water-repellency tests, which were previously unknown. The simple spray test seems to indicate only the effect of the waterproofing material and not the water-resistant properties of the fabric. It is, therefore, suitable only as a preliminary screening test to reject the entirely unsuitable materials. The hydrostatic-pressure test, on the other hand, overemphasizes the structure of the fabric but gives very little information about the water-repellent material. It is, therefore, most useful in eliminating unsuitable fabrics. The test, which most nearly duplicates actual conditions in use, is the impact-penetration test or the drop-penetration test. However, even here, serious discrepancies between laboratory results and actual use have been found. It is safest to report and compare fabrics on the basis of all three tests together.

Shrinkage

Shrinkage is the linear amount a fabric will contract warpwise or fillingwise when laundered. It is expressed as per cent of its original measurement. All fibers have some tendency to shrink, but this tendency is greatly increased if the fabric has been stretched in finishing. The introduction of the Sanforizing process, with its guaranteed maximum shrinkage, has brought shrinkage tests into prominence. Also, the development of new methods of decreasing the shrinkage of woolen and rayon goods has made shrinkage tests on these fabrics common.

Johnson has shown that shrinkage in cotton goods is nearly totally accounted for by the water, i.e., that the detergents used have little effect on the amount of shrinkage, although they may alter the rate of shrinkage or the time required for the full amount of shrinkage to occur. However, in spite of this fact the standard tests imitate real laundering as far as the chemicals which are used are concerned. It should be considered also that shrinkage is not complete in one laundering; although most of the shrinkage occurs in one treatment, a total of at least five launderings are necessary to get the total shrinkage.

All the standard tests involve the preparation of a sample by cutting a square of cloth and marking it either with indelible ink or with fine threads sewn into the fabric. The marks are made a measured distance apart both warpwise and fillingwise, three pairs in each direction. The samples are then laundered by varying methods which will be considered later.

After laundering and rinsing, the fabrics are dried on a horizontal surface and pressed while still damp by a flat-bed press, or by a flat-iron used without sliding to imitate a flat-bed press. The distances between marks are then measured and averaged and the resulting shrinkage expressed as per cent of original measurement or as inches per yard. Flat-bed presses suitable for use in these tests may be obtained in this country from the General Electric Company, Schenectady, New York, or from Franklyn Press Company, Syracuse, New York.

WASH WHEEL TESTING METHODS

The A.A.T.C.C. method for shrinkage of cotton fabrics, which is also the accepted method of the A.S.T.M., uses a sample at least 20 inches square, or better 20 inches long and the full width of the fabric, and a measured distance of 18 inches. The washing machine has a reversing wash wheel with a diameter of 20 inches. It has a capacity of 3 pounds of dry cloth and is fitted with a pipe for injecting live steam into the wheel for heating. Machines which answer these specifications may be obtained from Robert Ewing & Sons Company, Green Island, New York, or from Hurley Machine Company, 22nd Street and 54th Avenue, Chicago, Illinois. The machine is used with a full load and with solution equal to fifty times the weight of the load. The solution used is one of chip laundry soap such as will give *good running suds* and is made up from a stock solution of 1 pound of soap in 1 gallon of hot water. The load is put in, the machine started, and water and soap, at a temperature not exceeding 100° F., are added. The temperature is raised to 212° F. by injecting live steam. The steam is then shut off, and the machine is run for 40 minutes. The soap solution is drained off, water is run in, the temperature is raised to 140° F. and the machine is run for 5 minutes. The water is drained off, more water is run in, raised to 140° F. and the machine is run for 10 minutes. The water is drained off again and the load is run without further additions of any sort, to complete the full cycle of 60 minutes; during this time the machine should not be stopped.

The test for shrinkage of fabrics other than cotton (wool, silk, and rayon) is the same as above except that the washing is for 15 minutes at 100° F.; the first rinse is for 5 minutes at 100° F., the second rinse is for 10 minutes, and the wheel is stopped and drained and filled each time. This test duplicates very closely actual laundry conditions but requires special and rather expensive apparatus. It is suitable for laboratories doing a lot of shrinkage testing, but another method requiring no special apparatus would be desirable for laboratories making only occasional tests. The use of ordinary household washers has been found to be practicable in many cases with results as satisfactory as those with the wash wheel. The results, however, should first be checked against a standard wash-wheel test before adoption.

Launderometer Method

The A.S.T.M. method for use on silk and rayon woven goods uses a sample 12 or 13 inches square and measured distances of 10 inches. The soap solution used is 0.5% neutral soap, and the washing and rinsings are all done at 100° F. The machine used is a Launderometer, originally devised for obtaining the fastness of dyes to laundering. It consists essentially of a number of one-pint preserve jars held with their bases to a horizontal shaft 2 inches from the center of revolution, the shaft revolving at 40 to 45 revolutions per minute. The jars are maintained at the standard temperature of 100° F. ± 2° by a water bath. Each sample is placed in a jar with 300 cubic centimeters of soap solution and run for 15 minutes. The sample is removed from the jar, rinsed by being dipped in three changes of water, returned to the jar with 300 cubic centimeters of water and run for 5 minutes; it is then dried, pressed, and measured. This test requires only a Launderometer which can be used for other tests; the Launderometer may be obtained from Atlas Electric Devices Company, 363 West Superior Street, Chicago, Illinois.

Method Requiring No Special Apparatus

Williams has suggested a method of testing for shrinkage which requires no special apparatus. A sample 10 inches square with measured distances of 8 inches is used. The sample is first washed in warm water if any significant amounts of sizing are present. The sample is then immersed in a beaker containing 0.3% solution of soap in water at 40° C., and the beaker and contents are left in a warm place for at least 2 hours. The sample is removed, rinsed, squeezed as dry as possible in towels, allowed to dry while spread smooth on a pad of cotton sheeting, and ironed. Measurements are taken and the results reported as *ironed dry*. The sample is rewet, wrung out, ironed until dry, conditioned several hours, measured again, and the results reported as *ironed wet*. The ironed dry shrinkage is usually more than the ironed wet.

PERMISSIBLE SHRINKAGE

The permissable amount of shrinkage varies widely according to the use to which the fabric is to be put and the labelling which goes with it. A *guaranteed shrinkproof* or *100% shrinkproof* fabric should have no appreciable shrinkage; a *pre-shrunk* fabric should have only a small amount of shrinkage (1–2%). A Sanforized label implies not over 1% shrinkage in any direction and should be so understood. In general, we may say that, even without any label or claim, a shrinkage of more than 5% in either direction is excessive, except in the case of wool.

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Chapter 8

FABRIC TESTING—TACTILE AND DRAPING QUALITIES (HANDLE)

This quality of a fabric is now expressed only by the qualitative judgment of an expert who, on feeling, bending, and crushing a fabric in his hands describes it in such terms as *soft*, *harsh*, *boardy*, *lofty* or in some other terms which mean little to a non-expert and which do not allow accurate comparisons with other fabrics or with comparisons of the feel to other experts. Handle is a very complex mixture of properties which are unconsciously integrated or summed-up in the mind of the expert. The main, but probably not the only, factors in handle are:

1. Weight and density. Weight per unit area or per unit volume is considered. Weight has already been described in Chapter 5. Fabrics vary in weight and density from *compact* to *open.*
2. Surface friction, i.e., resistance to slipping either on the fingers or on another piece of fabric. Fabrics vary in surface friction from *harsh* to *slippery.*
3. Flexibility, i.e., ease of bending of a fabric. Fabrics vary in flexibility from *pliable* to *stiff.*
4. Compressibility, i.e., ease of squeezing. Fabrics vary in compressibility from *soft* to *hard.*
5. Resilience, i.e., ability to recover from deformation in any length of time. The deformation may be flexural, compressional, extensional, or torsional. Fabrics vary in resilience from *springy* to *limp.*

No one instrument will determine all of these but each one may be determined separately.

Surface Friction

Two methods of measuring surface friction are available:

1. Mercier method (inclined plane)
2. A.S.T.M. method (Friction Meter)

MERCIER METHOD

In the Mercier method, a block covered with a sample of the fabric to be tested is placed on an inclined plane covered with the same material. The angle between the inclined plane and the horizontal when the block just begins to slide is measured. Owing to a rearrangement of the fibers in the surface of the fabric, this angle decreases with successive slides; therefore, a number of slides is made until a constant angle is found. It may be shown that the coefficient of friction of the fabric on itself is equal to the tangent of the angle of slide. A low coefficient is desirable in the lining of coat sleeves, shoe linings, and sleeping garments, whereas it is undesirable in the backing of rugs.

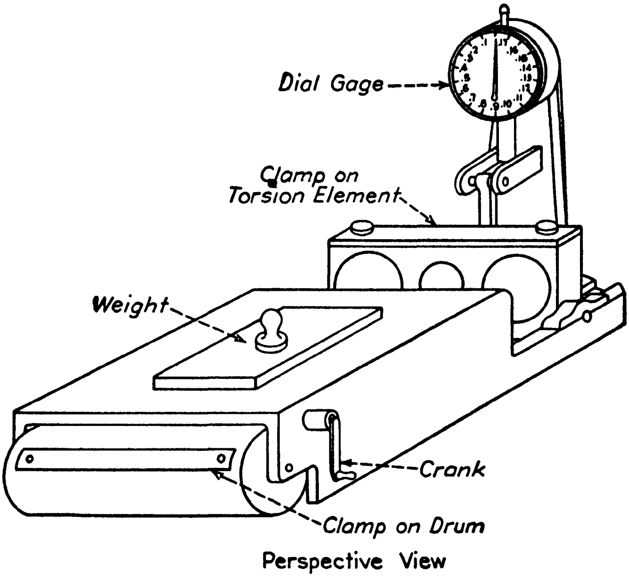

FIG. 24. Friction Meter. (*A.S.T.M.*)

A.S.T.M. METHOD

In the Friction Meter of the A.S.T.M. (Fig. 24), two pieces of the sample, 4 × 12 inches, are laid face to face on a polished horizontal surface under a one-pound weight having a polished bottom surface approximately 3 × 3⅓ inches. The bottom sample is attached to a drum so that it may be withdrawn at a speed of approximately 30 inches per minute. The upper sample is attached to a torsion spring with a dial which reads force in units of 0.02 pounds. On turning the drum, the lower sample is withdrawn and imparts a frictional drag to the upper sample, which is recorded on the dial. The initial force is high, as a result of static friction, but then becomes constant at a lower value during the latter half of the motion; this latter value is recorded.

Flexibility

The flexibility or the stiffness of a fabric may be measured in several ways:

1. By the thickness of a folded sample (MacNicholas and Hedrich method)
2. By the sag of a projecting strip of sample (Peterson and Dantzig method, Skinkle method, Peirce method)
3. By the length of a heart-loop (Peirce method)
4. By means of a Flexometer (Schiefer method)
5. By the moment of rotation (Saxl method)
6. By means of a Planoflex (Dreby or A.S.T.M. method)
7. By means of a Drape-Meter (Schwarz or A.A.T.C.C. method)

THICKNESS OF A FOLDED SAMPLE

The method of MacNicholas and Hedrich consists in folding a strip of fabric, 3 × 9 inches in size, back on itself and measuring the height of the fold when compressed by various loads. This method requires only a thickness gage with a variable load attachment.

SAG OF A PROJECTING STRIP OF SAMPLE

The method of Peterson and Dantzig is illustrated in Fig. 25. The strip of fabric is fed horizontally by rubber-covered rolls across a plane surface upon which is a metal bar at an angle (B′BC) of 45°. This bar is calibrated in millimeters, and the point C, at which the fabric touches the bar, is observed. The distance BC is a measure of the stiffness of the cloth.

A simpler method suggested by the author is to have a horizontal plane (AB) and a plane (BD) at 45°. The strip of fabric is placed on the horizontal plane with a metal rule above it, the end of the rule coinciding with the end of the fabric. The rule and strip are then moved to the right, the strip bending over and eventually touching the angular plane at which time the end of the rule is at B′. The distance BB′ read from the rule is then a measure of the stiffness. This apparatus may easily be made in the laboratory and is simple to operate.

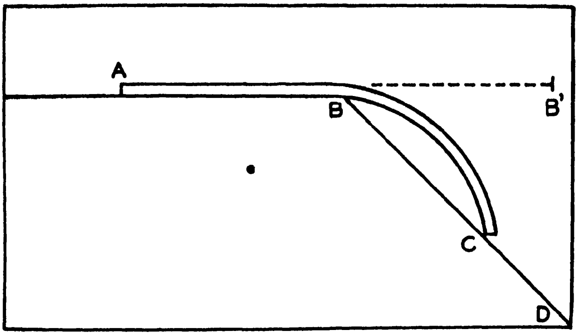

FIG. 25. Principle of Stiffness Determination

Peirce has described a more complicated method which, in principle, is the same as the one mentioned above except that a known length of cloth is allowed to bend over, and the angle B′BC is measured. The bending length may then be calculated:

$$C = L\left(\frac{\cos\frac{a}{2}}{8\tan a}\right)^{\frac{1}{3}}$$

when

C = bending length
L = length of the overhanging fabric
a = the angle $B'BC$

When a is 45°, the relationship becomes

$$C = 0.00154L$$

Peirce suggests that the resistance to bending which is felt by the fingers is due to the flexural rigidity which may be calculated:

$$G = WC^3$$

when

G = flexural rigidity
W = weight per unit area of the fabric

To compare the stiffness of materials of different thicknesses, the bending modulus may be calculated:

$$Q = \frac{KG}{d^3}$$

when

Q = bending modulus
d = thickness
K = a constant

LENGTH OF A HEART-LOOP

Peirce also suggests a method called the heart-loop method for use on very soft materials where the stiffness cannot be obtained by the previous methods. A strip of fabric of length L is folded back on itself and clamped so that it hangs in a heart-shaped loop (Fig. 26). If the fabric were very stiff, the length of the loop (m) would be 0.1337L, but if the fabric were completely limp (and the clamps were very thin), the loop would have a length (m) of 0.5L. If the length of the loop is measured, the stiffness is inversely proportional to the length (m) or the limpness is directly proportional to the length,

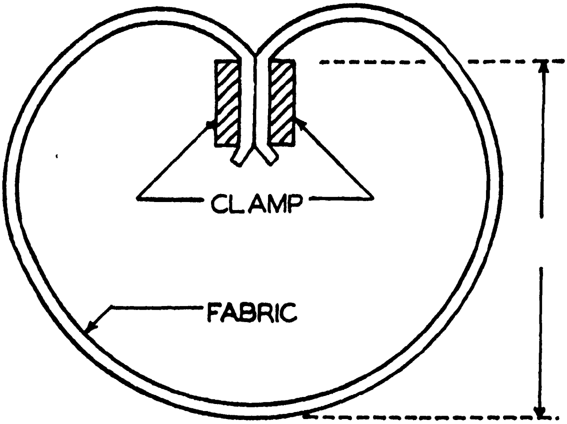

FIG. 26. Heart-loop Method for Stiffness

between the above limits. Calculations may also be made as in Peirce's other method:

Let

$$n = 0.1337L$$
$$S = m - n$$

Then

$$a = 32.85^\circ \times \frac{S}{n}$$

and

$$C = n\left(\frac{\cos a}{\tan a}\right)^{\frac{1}{3}}$$

USE OF A FLEXOMETER

Schiefer describes a *Flexometer* in which a pair of test specimens of standard dimensions are mounted in the vertical angles formed by two vertical intersecting plates, one fixed and the other movable on a spindle. The work done in folding the specimens to various angles between the plates is measured by the deflection of a calibrated spring.

MOMENT OF ROTATION

Saxl has devised a stiffness tester in which a strip of cloth is held by clamps at one end, the other end resting on the pan of a balance. The balance is set for a definite weight and the clamp is rotated, a pointer indicating the number of degrees until a balance is reached. This instrument, therefore, measures the force required to bend the fabric.

USE OF THE PLANOFLEX

The Planoflex (Fig. 27), developed by the A.S.T.M., measures the distortion angle through which a fabric may be distorted in its own plane without producing wrinkles in the fabric. The sample used is 3 ± $\frac{1}{16}$ inches in width and approximately 10 inches long. The apparatus has a moveable clamp, a fixed clamp, and a hinged shelf. The moveable clamp is attached to the fixed clamp by two strips so that it is able to move in an arc having a radius of 6 inches and always parallel to the fixed clamp. One end of the sample is fastened in the moveable clamp which is at zero and a clamp, 2 pounds in weight, which rests on the hinged shelf is attached to the other end of the sample. The hinged shelf is then released so that the sample is under tension, and the fixed clamp is tightened in place. The

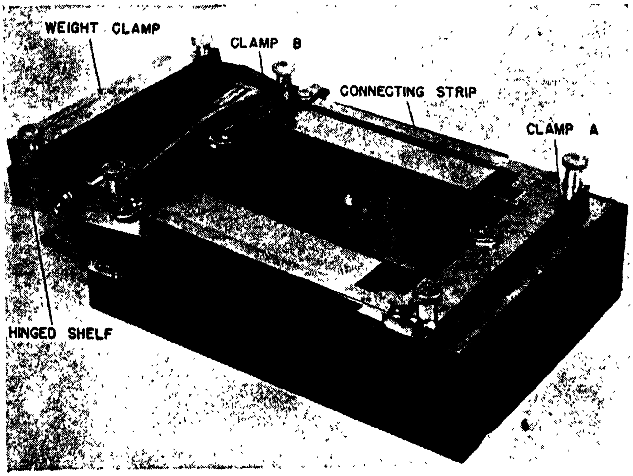

FIG. 27. Planoflex. (*A.S.T.M.*)

moveable clamp is then moved slowly, first to the left, and then to the right, until wrinkles appear in the sample. The sum of the left and right angles is recorded. This apparatus gives results which usually agree with those given by the Flexometer, the heart-loop method, and the judgment of experts.

USE OF THE DRAPE-METER

The A.A.T.C.C. has developed an apparatus called the Drape-Meter (Fig. 28) which measures not only stiffness but a combination of stiffness and other qualities. This combination of qualities is called *draping quality*. The apparatus consists of a semicircular disc to which the sample is attached and from which it hangs or *drapes*. The apparatus enables one to trace the curve of the cloth at different levels. It is obvious that an infinitely soft fabric would have a semicircular outline at all levels, whereas an infinitely stiff but elastic fabric would vary from a semicircle at the top to a straight line at the bottom; commercial fabrics would, of course, fall between these extremes.

Final details of manipulation are not yet worked out, but it is suggested that a specimen, 4.7 × 10 inches should be cut out with the short edge parallel to the direction to be tested. A pencil line should be drawn down the center of the specimen and an eight-inch length of one-half-inch cellulose tape should be attached to the top edge. The specimen should be attached to the semicircular disc so that the centers coincide and the pencil line hangs vertically. For a complete test, contour lines may be drawn at every six-centimeter level. The pointer should be brought to one side edge, the marker tapped to make a point on the paper beneath, the pointer moved in about 2 centimeters, and the process continued around the contour to the other edge. The points should then be connected with a colored line. For routine comparisons, the chord length at one level (between 9 and 18 centimeters) may be determined. The cloth should hang 10 minutes before a determination is made. The specimen should be tested face in and face out and the average taken.

The author has used a simplification of the Drape-Meter as follows: a sample of fabric, 100 × 250 millimeters is cut out, with the hundred-millimeter inch direction parallel to the yarns being tested. This sample is folded lengthwise, at one end gripped by a three-inch clamp at the folded end, and hung up by the clamp from a horizontal rod. At a distance of 100 millimeters downward from the edge of the jaws, the inside chord length of the fabric is read by a millimeter scale; thus, each millimeter represents 1% of the fabric width. After the measurement, the fabric may be completely folded, placed between

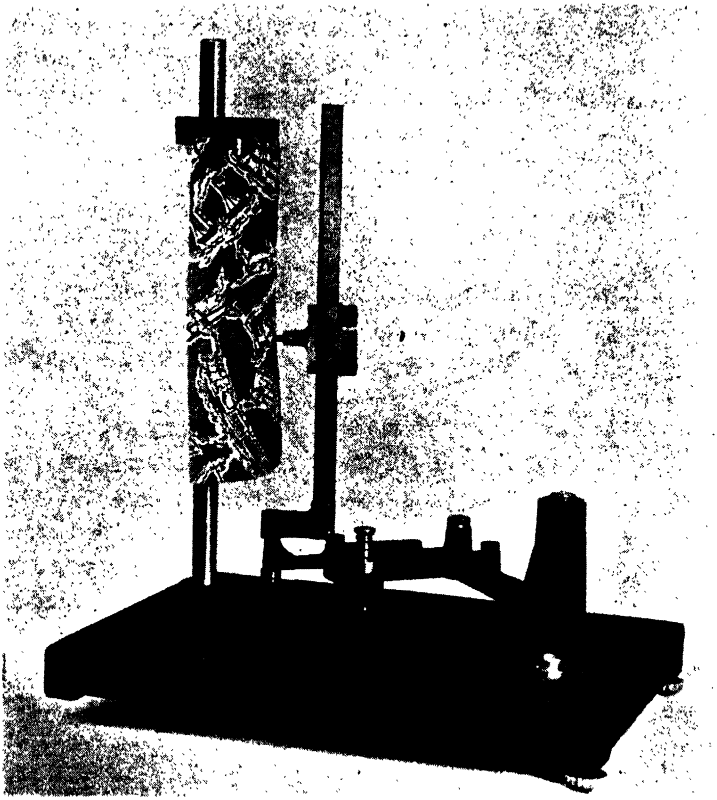

FIG. 28. Drape Meter

two flat surfaces with a standard weight for a standard length of time and again measured. The difference between the two readings is a measure of the resilience, and the first reading is a measure of the stiffness in the hundred-millimeter direction.

Peirce has shown that the stiffness of a fabric as a whole is the geometric mean of the stiffness of the warp and the filling, that is, the stiffness warpwise and the stiffness fillingwise are measured, multiplied together, and the square root of the product obtained.

Compressibility

The compressibility or hardness of a fabric may be measured by:

1. Variable load thickness gage (Peirce)
2. Compressometer (Schiefer)
3. Compression Meter (Dreby or A.S.T.M.)

Variable Load Thickness Gage

If the spring is removed from a standard thickness tester and a light platform, to hold weights, is fastened on the top of the presser-foot rod, we may measure the thickness of the fabric under varying loads. If the thickness is plotted against the load, a curve similar to a, Fig. 29 is obtained, consisting of a small curved portion and then an approximately straight line. Peirce suggests the measurement of the thickness (T_1 and T_2) at two loads (L_1 and L_2) on the straight-line portion of the curve; the hardness of the sample (H) is then given as:

$$H = \frac{L_2 - L_1}{T_1 - T_2}$$

Use of the Compressometer

Schiefer describes the *Compressometer* as a combination of two thickness gages mounted with a rack and pinion to move vertically through a helical spring. The lower thickness gage reads the thickness of the fabric. The upper thickness gage reads the deflection of the spring, which, combined with the calibration of the spring, gives the load on the fabric. Schiefer defines the compressibility of a fabric as the rate of decrease of the thickness at a pressure of 1 pound per square inch divided by the thickness at the same pressure, using the straight line in a, Fig. 29 to get the slope which is the rate of decrease of thickness, or using the same symbols as before:

$$\text{Compressibility} = \frac{T_1 - T_2}{T_1(L_2 - L_1)}$$

If the Compressometer is used with a pile fabric, a curve similar to the upper one of c, Fig. 29 is obtained; in this, AB represents the thickness of the pile, L is the crushing load, and BC represents the bending of the pile.

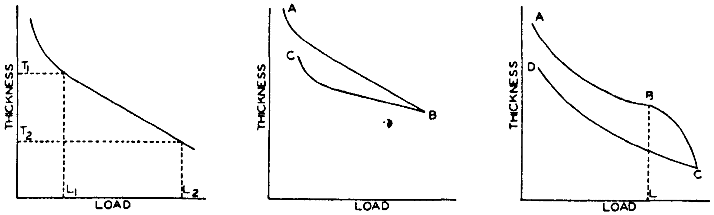

FIG. 29. Typical Thickness-Load Curves for Fabrics

a. Ordinary fabric-increasing load

b. Ordinary fabric-increasing and decreasing load

c. Pile fabric-increasing and decreasing load

USE OF THE COMPRESSION METER

The compression meter (Fig. 30) of Dreby, which has been adopted by the A.S.T.M., consists of a circular base plate over which a sample 6 × 6 inches, is stretched with a hoop to keep it taut. Above the sample is the meter itself which has a diaphragm 3 ± 0.005 inches in diameter in contact with the sample, a cavity above it filled with liquid, a micrometer piston to force liquid into the cavity and capable of being read in units of 0.0001 cubic inches, and a manometer graduated from 0.05 to 0.45 pounds per square inch. With the meter in place, the piston is advanced until the pressure reads 0.05 for 30 seconds. The volume is then read on the micrometer of the piston. The pressure is then raised until 0.45 is maintained for 30 seconds and the second volume is read. The difference in the two volume readings is recorded; this is the total volume compression for a circular area 3 inches in diameter and with a pressure increase from 0.05 to 0.45 pounds per square inch.

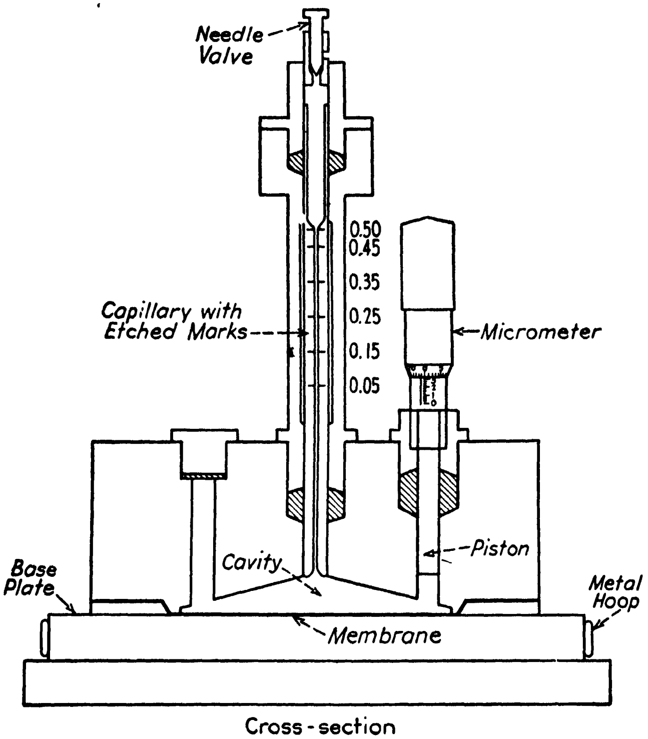

FIG. 30. Compression Meter. (*A.S.T.M.*)

Resilience

The resilience of a fabric is obtained by measuring the deformation of the fabric on increasing load, then measuring the deformation on decreasing the load. The two curves are plotted, and the area under each curve measured by a planimeter or by taking a number of measurements:

$$\text{Resilience} = \frac{\text{area under curve of decreasing load}}{\text{area under curve of increasing load}} \times 100$$

A simpler method consists in measuring the deformation (D_1) under a light load (L_1), then increasing the load to L_2 and measuring the deformation (D_2); the excess load is then removed to obtain load L_1 again and the new deformation (D_3) is measured. Then,

$$\text{Resilience} = 100\,\frac{D_2 - D_3}{D_2 - D_1}$$

This determination may be applied to many of the stiffness or hardness determinations already described. The Haven method for fiber resilience described in Chapter 3 may be used for fabrics also. The MacNicholas and Hedrich method for stiffness may be run with decreasing loads and the resilience measured. Schiefer's Flexometer and Saxl's apparatus may also be used to determine resilience. A variable load-thickness gage or Schiefer's Compressometer will give curves as in band c, Fig. 29 and may be used to measure resilience. The Compression Meter also may be used to measure resilience.

Amick has described a method of measuring the crease resistance of fabrics which really measures the resiliency of the fabric after creasing. Strips of fabric are cut out 4 centimeters long and 1 centimeter wide, warpwise and fillingwise, and are allowed to condition, after which they are handled only with forceps. Each strip is folded in half (2 × 1 centimeters), placed on a glass plate, and a weight of 500 grams is placed on it for 2 minutes (by stop watch). The weight is removed, the strip is placed on a taut wire for 2 minutes, and then the distance between the ends of the strip is measured. The distance in centimeters divided by 4 and multiplied by 100 is the per cent resilience of the sample.

The handle or draping quality of cloth is of great importance in the application of starch to fabrics, in the calendering of cotton, in woolen and worsted finishing, and in the determination of the crease resistance of fabrics. The introduction of the synthetic resins for creaseproofing fabrics and the introduction of various softeners for use in finishing fabrics also make it necessary to give a quantitative value to the various factors which affect the handle of the cloth.

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Chapter 9

FABRIC TESTING—SERVICEABILITY, WEAR, ABRASION

Serviceability, wear, and abrasion are terms which are often used interchangeably, but in reality there is a distinction.

Serviceability

Serviceability of a fabric is its length of life up to its end of usefulness which occurs when it becomes deficient in one necessary property. In clothing, for example, the end of service generally is reached when, due to color fading, shrinkage in laundering, or bagging at knees or elbows, the garment no longer has a presentable appearance; clothing seldom wears out, although some examples of actual wear may be found. The serviceability of tent cloth is determined by its waterproofness; the serviceability of canvas belting or shoe lining, on the other hand, is determined by actual wear. Serviceability, then, is indeterminate and depends on the length of time or amount of use a fabric may endure before its most desirable property decreases to the point where the fabric is no longer useful.

Wear

Wear is the amount of deterioration of a fabric due to breaking, cutting, or removal of the fibers. In certain cases such as the belting or shoe linings quoted above, wear and serviceability are probably the same, but in most cases they are distinct. Wear is also an indeterminate quality because conditions of wear vary so much and also because we do not know quantitatively the effect of the different factors involved in wear. Qualitatively, the following are most likely the factors causing wear:

1. Direct force on the fabric. This never occurs in normal wear but only under abnormal stress.
2. Impact effects. These are important only in floor coverings.
3. Flexing, or friction between fiber and fiber and between yarn and yarn due to bending of the fabric. This is ordinarily unimportant because it results in very slow wear; in the case of sized or coated fabrics, however, it is very important.
4. Abrasion
   - a. Friction between cloth and cloth. This occurs only locally, such as the rubbing of the sleeve on the coat.
   - b. Friction between the cloth and external objects. This is probably the most important factor.
   - c. Friction between the fibers and the dust or grit in the fabric, resulting in the cutting of the fibers. The importance of this factor depends upon the character of the fabric or the use to which it is put and the frequency and thoroughness of cleaning.

Abrasion

From the above, it can be seen that abrasion is the most important factor in wear in most cases and that abrasion consists of friction between the cloth and some other material. For purposes of testing, this material must be quite rough so as to give a quick result, but the selection of this rough abrasive may be contrary to the ordinary conditions of use of the fabric. This should be considered in evaluating the results of abrasion tests.

The results obtained on an abrasion tester are comparative only. In many cases, the order of resistance of fabrics to abrasion is also the order of wear, as far as we can tell; but in many other cases the order is different. The abrasion test must not be considered as imitating wear because wear is such a variable process and we have no means of measuring it. The abrasion test may not be considered a measurement of serviceability. The abrasion test is simply a test of the quality of the fabric as to its resistance to a combination of flexing and cutting of the fibers, and its results should be considered in connection with other tests and not as a single criterion of the suitability of a fabric for a given use. The tester should continually keep in mind the fact that abrasion is not the only factor in wear, and wear is not the only factor in serviceability.

MAIN FACTORS

The main factors in testing abrasion, in addition to the temperature and relative humidity of the atmosphere which should always be at standard conditions, and their general effects are:

1. Character of the motion
   a. Rotary
   b. Reciprocating

In general, reciprocating motion is better because the effect of abrasion may be studied on the warp and filling separately.

2. Nature of the abradant
   a. The sample itself
   b. A standard cloth, usually canvas
   c. A steel surface
   d. Silicon carbide, emery cloth, or emery paper

If the sample itself is the abradant, a long period of abrasion is required and different cloths are not comparable with each other. If a standard cloth is used, variations are very apt to exist so that results obtained at one time cannot be compared with results obtained at another time. A steel surface is also very variable not only from one machine to another but in the same machine after different periods of wear. The use of silicon carbide or emery cloth is now almost universal; although admittedly unlike real wear, it gives rapid results and can be obtained quite uniform.

3. Pressure of the abradant on the sample

This effect is very great, usually a linear relationship exists between the pressure of the abradant and the amount of abrasion. The pressure, therefore, must be standardized.

4. Tension on the sample

This effect is also great and the tension must be standardized

5. Removal of lint
   a. By a vacuum attachment
   b. By running the machine at high speed so that the lint is thrown out
   c. By rocking the abradant to brush out the lint

Accumulation of lint in the abradant or in the sample tends to clog the machine and reduce the amount of abrasion.

6. Determination of end point or amount of abrasion
   a. Number of strokes required to wear a hole
   This appears the most logical method but actually is affected by so many variables that it is rather indeterminate; it is, however, frequently used.
   b. Loss in strength after a definite number of rubs. This is used in most cases and seems to give most satisfactory results. A large enough area must be abraded, then, to give sufficient strength tests to obtain a satisfactory average.
   c. Loss in thickness after a definite number of rubs. This is used only on felts, carpets, and napped fabrics where considerable abrasion may occur before any loss in strength takes place. In general, thickness determinations are unsatisfactory.
   d. Loss of weight after a definite number of rubs. This method is used on heavy plushes and deep-pile goods where thickness determinations are least satisfactory; it is not used on ordinary woven goods because of the possibility of the cloth picking up pieces of lint or abradant.

e. Change in permeability, compressibility, heat insulation, water absorption, or other properties. These are used only in very special cases.

ABRASION MACHINES

Numerous abrasion machines have been described in the literature and for most of these the reader is referred to the survey by Ball of the machines commercially available; these include the following machines together with the manufacturer or dealer: Amsler Wear Tester sold by Herman A. Holz, New York; M.I.T. Abrasion Machine made by Hamblett Machine Co., Lawrence, Mass.; Perspirator by Industrial By-Products and Research Co., Philadelphia, Pa.; Shawmut Wear-Testing Machine made by Shawmut Engineering Co., Boston, Mass.; U. S. Testing Co., Inc., Abrasion (Wear Test) Machine made by United States Testing Co., Inc., Hoboken, N. J.; Wyzenbeek Precision Wear Test Meter made by Wyzenbeek and Staff, Inc., Chicago, Ill.; Schopper Abrasion Tester sold by Testing Machines, Inc., New York. The most satisfactory are the M.I.T. model described by Haven and Taber *Abraser* sold by the Taber Instrument Co., North Tonananda, New York.

M.I.T. Model

In considering the M.I.T. model, we might first note how it compares with the factors given above.

1. The sample is a strip of cloth 26 inches long and 6½ inches wide which is abraded by a longitudinal reciprocating motion so as to abrade 18 inches of sample; 1½ inches at each end are abraded unevenly, the 15 inches in the middle are abraded at uniform speed.

2. The abradant for general use is No. 000 emery cloth; for carpets and materials which are doped or coated, No. 00 garnet paper is recommended.

3. The pressure of the abradant on the sample may be varied. It is suggested that a pressure equal to the number of ounces per square yard of the fabric be used.

4. The tension on the sample may be varied from 1 pound up. It is suggested that tensions be as follows:

Light fabrics	up to 7 ounces per square yard	1½ pounds
Medium fabrics	7–15 ounces per square yard	2 pounds
Heavy and coated fabrics	above 15 ounces per square yarn	2½ pounds

5. Removal of lint is accomplished by having the abradant free to rock. This rocking occurs with a snap at each reversal of direction of motion and throws off any lint formed by the previous stroke.

6. The amount of abrasion is determined by giving the per cent loss in strength after a definite number of rubs. A revolution counter gives the number of double rubs and the size of the sample is such that ten strip or four grab tests may be made on the abraded surface.

In addition to the above factors, this machine includes flexing of the cloth over a roller moving in ball bearings. The diameter of the roller controls the amount of flexing and the following sizes are suggested:

Light fabrics	up to 7 ounces per square yard	¼ inch in diameter
Medium fabrics	7–15 ounces per square yard	½ inch in diameter
Heavy fabrics	above 15 ounces per square yard	1 inch in diameter

A horizontal roller in ball bearings is mounted in the frame and over this is draped the sample. One end of the sample is clamped to a block which slides in vertical guides; the other end is attached to a dead-weight tensioning device. The sliding block is driven by a connecting rod and crank and by means of a heart-shaped cam is given 18 inches of linear motion of which the first 1½ inches are accelerating, the last 1½ inches are decelerating, and the middle 15 inches are uniform speed. The normal speed of operation is 55 to 60 revolutions per minute of the cam, the source of power being a one-half horsepower motor equipped with a rheostat and a speed-reducing device.

The abradant is stretched flat on a rocking plate and is fastened by screws. In the rocking plate, at each end, are two posts loosely fitting the top bars of the machine. In the machine, the yarns, which are perpendicular to the direction of motion, receive the most wear, so two tests are usually made, one on warp and one on filling. To measure the tendency of a cloth to *wear shiny*, a glass rod may be used instead of abradant and the increase in gloss measured with a photometer.

Taber Machine

The Taber Instrument Company has introduced an apparatus which they call an *Abraser*. A small circular piece of fabric is abraded by the action of two wheels which rub back and forth and criss-cross their paths so that the abrasive action

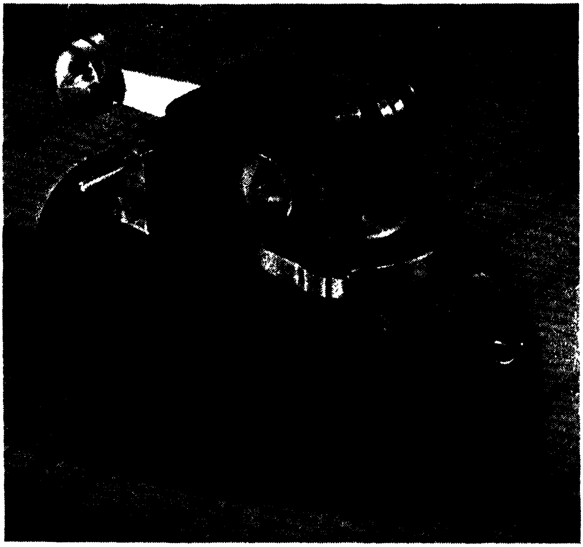

FIG. 31. Taber Abraser. (*Courtesy of Taber Instrument Corporation*)

occurs at all angles to the weave (Fig. 31). Provision is made for maintaining a constant pressure on the wheels and for counting the number of rubbing cycles to which the fabric is subjected. The abraded portion is in the form of an annular area and is not large enough to permit measuring strength. Visual comparison at the end of a definite number of cycles may be used for comparing fabrics or the number of cycles to form the first hole may be noted; this last method, as we have already seen, is not too precise. The Taber apparatus is easy to use and gives results quickly. It is satisfactory for comparing the same fabric before and after a treatment or for comparing different lots of the same fabric, but it does not give a numerical result, except for loss of weight. However, this result is questionable. For general use, the Abraser may be used for preliminary sorting of fabrics and eliminating the poorest. Further abrasions may then be conducted on other apparatus to get precise numerical results on the best fabrics.

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Chapter 10

STRENGTH AND STRETCH TESTING OF FIBERS, YARNS, AND FABRICS

Introduction and Definitions

The determination of the strength of a textile material gives us very useful information concerning that material, but a word of warning as to the interpretation of the results must be given. The strength test as an imitative test is of no value. In actual use, a textile material is not subject to a slowly and steadily increasing load as is used in the strength test but rather to abrasive action and smaller repeated loads. Strength as a measure of uniformity is a very useful test, since a change in any physical property or a change in the chemical composition of a textile material will nearly always result in a change in strength; even here, however, a warning must be voiced, as strength tends to give minimum rather than average results. To explain this, consider the measurement of twist in a yarn, we untwist 10 inches of yarn and measure the total number of turns. The average twist of the 10 inches of yarn is obtained in this way. If we break the same 10 inches, the force measured is not the average strength of that 10 inches, but the strength of the weakest spot in that length of yarn.

The principal value of the strength test is its use to measure the quality of the textile material, which is dependent upon the qualities of the constituents of the material and the quality of the work done upon it. For example, the quality of a yarn is dependent upon the quality of the fiber and the spinning operation; the quality of a cloth is dependent upon the quality of the fiber, the spinning operation, the weaving operation, and the finishing operation. It follows, then, that strength is the result of several variables and is rather complex. Wherever possible, then, strengths should be measured in comparison with other samples which differ only in one aspect from the unknown sample.

Stretch, especially if it is of the elastic type, is desirable in some cases; knitted goods, for example, are meant to be tight fitting and to yield readily to forces. In general, however, stretch is undesirable because the fabric is meant to keep its shape and dimensions. The combination of stretch and strength gives the work necessary to break the sample and is a measure of the toughness of the material, which is more nearly related to wearing ability than either strength or stretch alone. Consider as two extremes ramie, which has a high strength but low stretch, and wet rayon, which has a high stretch but low strength. The product of strength and stretch in both cases is low; both are lacking in toughness. Other fabrics may have less strength than ramie and less stretch than wet rayon, but the product of the two is greater and consequently they wear longer and better.

Unfortunately, the nomenclature of strength and stretch testing is not yet standardized, so we must first define the terms which we are about to use.

A *stress* is the force or load acting upon a sample; a *strain* is the change in dimension resulting from the stress. Stresses may be tensile stresses which result in an increased length of sample, compressive stresses which result in a more compacted sample, and torsional stresses which result in a twisted sample: in this chapter we shall consider only tensile stresses.

Breaking stress, *breaking load*, *breaking strength*, and *tensile strength* all refer to the force acting upon a sample at the time of failure; this is usually the maximum force applied.

Bursting strength is the force applied to break a fabric when applied at right angles to the fabric and uniformly distributed over a given area.

Tenacity is the breaking strength of a sample expressed in units of force per unit of cross-sectional area of the sample. In the case of rayon or silk it is the breaking strength in grams per denier.

Stretch or *extension* is a strain resulting in a greater length. Two types of stretch exist, either or both of which may be present in a given test, elasticity and elongation.

Elasticity is recoverable stretch. *Elongation* is nonrecoverable stretch. If a sample 10 inches long is stretched until it is 13 inches long, it has a stretch of 3 inches or 30%; if we now release it and it returns to a length of 11 inches, if has an elasticity of 2 inches or 20% and an elongation of 1 inch or 10%. Stretch, elasticity, and elongation may be given in units of length but are generally expressed as per cent of the length of sample broken, i.e., the length of the sample between the gripping jaws of the testing machine, called the gage length.

Elastic limit is the load below which the sample shows elasticity and above which it shows elongation.

Yield point is a wholly theoretical figure and is the load at which elongation would begin provided the material had no elasticity; this value cannot be directly measured but may be obtained from a stretch-load curve.

Elastic limit and yield point are usually determined only on rayons and silks and are expressed in grams per denier.

Another stretch phenomenon is that of *creep*. If a fiber, yarn, or fabric is held under a constant load for a long period of time, it develops a stretch which, when the load is removed, only slowly disappears. If left without a load for a long period of time, the sample gradually loses part of this stretch. This creep is obviously not the ordinary kind of elasticity, but neither is it permanent elongation. Creep does not show up on strength and stretch testing machines because of the short time interval during which the load is applied and so will not be considered further in this chapter.

Strength Testing Machines

Based on the method of loading, strength-testing machines may be classified as:

1. Type which is manually loaded by weights, chains, electromagnet, or trickling water
2. Pendulum type
3. Electric strain gage type
4. Constant specimen-rate-of-load type
5. Impact or ballistic tester
6. Bursting tester

Manually Loaded Strength Testers

The manually loaded type of strength tester is obsolete except for a few types of single fiber testers among which are the following:

O'Neill tester
Bowman tester and Matthews tester
Barratt tester
Krais (Deforden) tester

The O'Neill single fiber tester consists essentially of a pair of clamps for the fiber. The top pair of clamps is fixed, and the lower pair is attached to a floating *bob*. With the fiber in place, water is run out of a glass cylinder in which the bob is floating until the fiber is straightened, then a graduate is put in place. Water is run out of the cylinder into the graduate until the fiber breaks. The amount of water required to break the fiber indicates the breaking strength of the fiber. If testing is carried out at a constant humidity, calcium chloride solution of the same partial pressure as the atmosphere is used instead of water.

The Bowman and the Matthews fiber testers use an analytical balance in which one pan is replaced by the jaws of the fiber tester. Loading is done by placing weights on the other pan or using a chain for weighting the other arm of the balance. Barratt's apparatus is also an equal-arm balance in which the loading is done by an electromagnet. Krais' apparatus is an equal-arm balance in which the loading is done by running water into a cup on one side of the balance.

Pendulum Strength Testers

The pendulum tester is the type in common use for all textile materials. The specimen is strained by a uniform movement of a pulling jaw or clamp to which one end of the specimen is attached. The other end of the specimen is held by a jaw or clamp attached to a pendulum. The pendulum is displaced from its normal vertical position by a pull on the specimen, and the amount of displacement indicates the load. The pendulum is lightly ratcheted to a notched sector so that after the specimen breaks, the pendulum (and the pointer geared to it) will remain stationary at the maximum load.

The pendulum tester consists of three main parts:

1. Straining mechanism
   a. Constant speed motor, gear train, nut, and screw; or
   b. Cylinder and piston, with hydraulic pressure
2. Jaws or clamps holding the specimen
3. Loading and recording mechanism
   a. Chain
   b. Drum
   c. Pendulum arm and weight
   d. Pointer and scale
   e. Notched sector and ratchet
   f. Autographic stretch-load recorder.

The tester may be operated by a motor or by hydraulic pressure. Jaws are available for various tests on cloth or yarn. The pointer may be attached to the drum and indicate on a circular scale, or it may be attached to the end of the pendulum and indicate on a scale engraved on the notched sector. The autographic recorder (not always present on a strength tester) consists of a flat plate moved vertically by an attachment to the lower jaw and a pen which moves over a guide horizontally by attachment to the pendulum; it, therefore, records stretch vertically and load horizontally. Figures 32 and 33 show the principle and the parts of a motor-driven strength tester with an autographic recorder as used on cloth. This is the most common type of tester with a capacity up to 500 pounds used in this country. Older forms were operated by hand turning, but these gave very variable results and have been abandoned. Machines are often so made that removing one weight from the pendulum will convert it to a machine of lower capacity, usually one-half the original capacity.

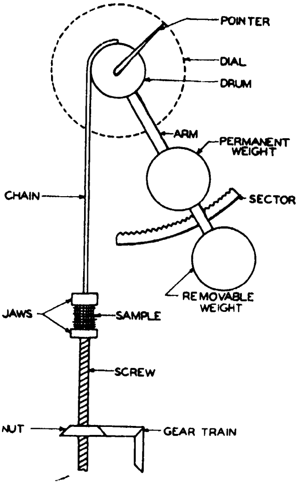


FIG. 32. Principle of Pendulum Strength Tester

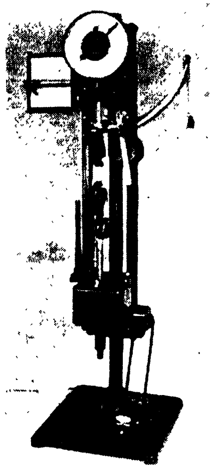


FIG. 33. Pendulum Strength Tester with Autographic Recorder. (*Courtesy of Scott Testers, Inc.*)

Some of the variations in pendulum testers are:

1. Hydraulic type mentioned above.

2. Rubber testers provided with a very long screw because of the enormous stretch.

3. Serigraph testers made especially for silk fiber, yarn, and cloth testing. These are made a little more finely than the ordinary tester and are always provided with an autographic recorder.

4. Horizontal testers. These are the same in principle as the ordinary tester but of high capacity (800, 1000, 2000 pounds).

5. Single-strand testers (Figure 34). These are similar in principle to the ordinary tester but of much lower capacity, never over 50 pounds, and usually reading in grams or ounces.

6. Moscrop single strand tester. This is designed to carry out a number of single-strand tests at once, recording the results on a chart. This is not as accurate as the ordinary single-strand tester but is good for showing variations in strength.

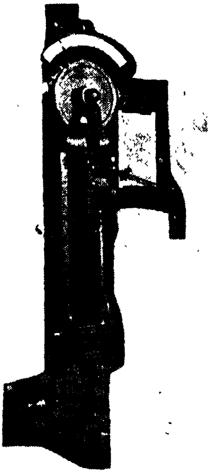


FIG. 34. Pendulum Single-strand Tester with Autographic Recorder. (*Courtesy of Scott Testers, Inc.*)

7. Fiber strength testers of the pendulum type. These are very low-capacity machines.

Before use, the strength tester should be zeroed and calibrated. It is zeroed by lifting the ratchet and letting the pendulum hang vertically; the pointer is then set at zero on the scale. To calibrate, a weight is rested on the lower jaw and attached by a wire or cord to the upper jaw. The machine is started and the weight comes on the upper jaw at the same rate of speed as the stress on the specimen. The scale should be calibrated at a number of points on its range and a correction table or chart prepared.

Assuming that the samples are tested at a constant relative humidity and temperature, the variables due to the machine itself, which affect the apparent strength of the specimen, are:

1. Friction and inertia errors. These are a function of the construction and care of the machine and have been shown by Haven to be not over $2\frac{1}{2}\%$ at the most. If the machine is well made and kept cleaned and oiled, this error is negligible and, at any rate, would be constant.

2. Form of the jaws. The jaws should be so made as to avoid cutting of the specimen. The standard types of jaws will be taken up later. The A.S.T.M. has laid down the following general rules regarding clamps or jaws of strength-testing machines:

The jaws shall have metallic gripping surfaces which are smooth, flat, and parallel.

The dimension of the clamp parallel to the direction of application of the load shall be 1 inch and in the direction perpendicular to the load shall be as specified for each test.

All edges which might cause a cutting action on the specimen shall be rounded off to a radius of not over $\frac{1}{64}$ of an inch.

One clamp of each pair shall be fixed in position, the other shall be hinged or swivelled and the clamping action may be by either screw or cam.

3. Gage length or distance between jaws. It has been shown that breakage of textile materials is mostly due to slippage of the fibers. If the gage length is increased, more slippage can occur and the breaking strength will be less. Another factor is the fact that in a longer length of specimen there is more chance of a weak spot occurring and giving a lower breaking strength. Still another reason is that a longer gage length means more stretch of the specimen so that a longer time is required to reach the breaking point; the rate of loading is, therefore, slower and a decrease in strength results. Following are the relative breaking strengths for different materials at three gage

lengths. These data are for illustration only and not for correcting results from one gage length to another.

	Gage Lengths 1 Inch	 3 Inches	 6 Inches
Rayon yarn	100	100	93
Silk yarn	111	100	93
Worsted yarn	125	100	88
Cotton yarn	111	100	92

4. Speed of the lower jaw. The slower the lower jaw moves, the more time there is for the fibers to slip and the lower the strength reading will be. Standard speeds will be given later. The following will illustrate the effect of jaw speed on the relative strengths of some samples.

	Jaw Speeds		
	6 Inches per Minute	12 Inches per Minute	18–20 Inches per Minute
Rayon yarn	97	100	104
Silk yarn	90	100	103
Worsted yarn	97	100	103
Cotton yarn	99	100	104
Cotton duck	99	100	102
Wool felt	97	100	101

5. Capacity of the strength-testing machine. A fabric of low strength on a high-capacity machine gives a high breaking strength because the time required to break the sample is short and, therefore, less slippage of fibers can occur. The following data gives the effect on a cotton cloth.

Machine Capacity Pounds	Strength Pounds	Time to Break Seconds	Relative Strength
150 pounds	65.8	5.25	100
300 pounds	67.8	3.85	103
400 pounds	69.5	3.10	106
800 pounds	75.4	2.55	114

It is generally specified that the maximum swing of the pendulum shall not be over 45°. It is also specified that the lower 20%

of the capacity (first 9° of swing) shall not be used. This often limits the machine which may be used on a given sample to one particular capacity. However, often several machines might be used and would give different results.

6. Stretch of the specimen. A specimen with a large amount of stretch takes up the load slowly and gives a lower breaking strength.

It will be noted that the variables mentioned under 4, 5, 6 and part of number 3 are all dependent upon the time of load application or rate of loading and that these variables would be eliminated if the rate of loading were constant, the method of doing this will be discussed later under another heading.

TESTING CLOTH

Cloth samples may be tested in one of three ways:

1. By strip samples
   - a. Cut strip
   - b. Ravelled strip
2. By grab samples

Cut strip samples are prepared very simply by cutting specimens 1 inch wide and 6 to 7 inches long. These samples have the disadvantage that the lengthwise yarns at the edge tend to spring away from the center yarns and, having a large amount of crimp, do not take up their share of the load. Cut-strip samples, therefore, are used only on heavily sized or coated fabrics and not on ordinary fabrics. Ravelled strip samples are cut out 6 to 7 inches long and 1¼ inches wide if there are fifty or more yarns per inch, or 1½ inches wide if less than fifty yarns per inch. After cutting out, the specimens are ravelled down to a width of 1 inch by removing approximately the same number of lengthwise yarns from each side. This is done so that the edge yarns may be supported and held in place by the cross yarns almost as firmly as the middle lengthwise yarns. In the case of both types of strip samples, the clamps used are 1½ inches or more wide, the gage length is 3 inches, and the speed of the lower jaw is 12 ± ½ inch per minute. The results of specimens which slip in the jaws or break at the edge of the jaws should be discarded. For this reason, it is a good idea to always prepare a few more samples than are actually called for in the test specifications.

Grab-test specimens are cut out 4 inches wide and 6 inches long, with the longer dimension in the direction of the yarns to be tested. The front clamps should be 1 inch wide and the back clamps 2 inches wide, the gage length should be 3 inches, and the speed of the lower jaw should be 12 ± ½ inches per minute. In this case also, the results of specimens which slip or break at the jaws should be discarded. An effort must be made to grip the same yarns by both upper and lower jaws, to facilitate this, a line may be drawn along one thread and the sides of the jaws placed on this line.

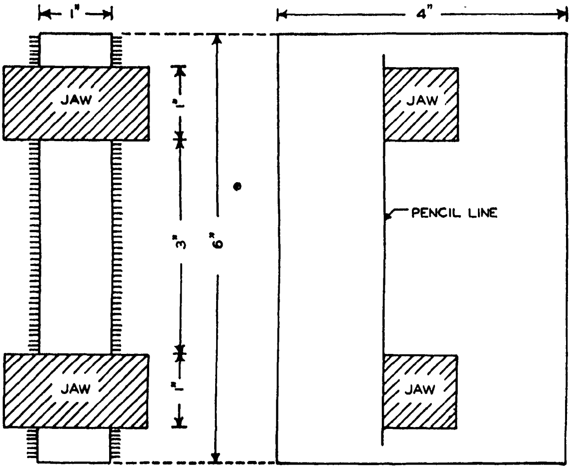


FIG. 35. Ravelled Strip and Grab Cloth Samples

In cutting out samples of fabric for test, at least five tests must be made on the warp and five on the filling. Samples should be taken as shown in Fig. 36. If ten samples are desired (much better than only five samples), the sampling is repeated on the other side of the center line of the cloth and further along the piece.

Strip tests are more reliable and accurate than grab tests but grab tests are much faster to carry out and are especially good on fairly open fabrics. Strip-test results are directly proportional to the number of yarns in the width. Therefore, strip tests are good tests of the component yarns. Grab-test results do not show as good a relationship between strength and number of yarns gripped but are satisfactory for ordinary testing. The A.S.T.M. uses grab tests on all fabrics for control work, but the strip tests are better for research work. The higher results on the grab strength are due to the reinforcing effect of the side yarns not directly gripped by the jaws.

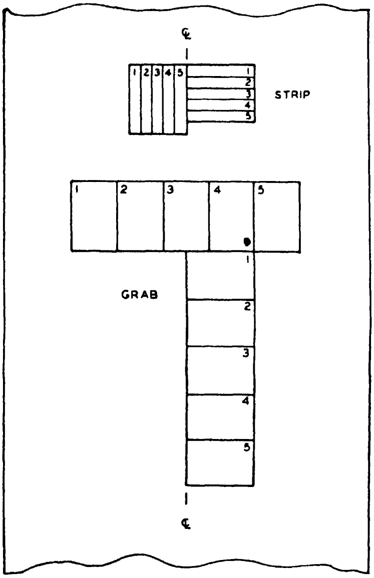


FIG. 36. Sampling of Fabric for Strength Tests

LaRose has suggested the use of a *half-grab* sample. This is cut out 6 inches long and 2 inches wide. Then yarns are unravelled along one long edge for about $\frac{1}{4}$ inch in from the edge.

The sample is placed in the grab test jaws with the ravelled edge along the side edges of the jaws. The results obtained are intermediate in value between strip and grab tests since the reinforcing effect of the outside yarns are exerted only on one side of the jaws. These samples are as easy to prepare as grab samples (if a line is required on the grab samples) and give perfect alignment of the yarns in the jaws. They are much easier to prepare than strip samples and give a lower variation in results than either strip or grab samples.

Woven tapes are tested in full width with the same gage length and jaw speed as above; the filling is not generally tested.

TESTING YARN

Yarns are tested in one of the following ways:

1. Skein test
2. Single-strand test
3. Serigraph test

The selection of the method to be used in any particular case depends on the purpose of the test, each method having certain advantages and disadvantages. The A.S.T.M. specifies the single-strand test as the preferred test with the skein test as an alternate method.

For the skein test, the yarn is wound on a 1½ yarn reel. The yarn is drawn from the top of a bobbin, cop, cone, or small flanged spool and drawn from the side of a parallel tube or large flanged spool; the ends of the skein are tied in a square-knot. In the case of spun yarns, a 120 yard skein is used; in the case of filament yarns, skeins are wound as follows:

Denier of Rayon	*Number of Turns*
Finer than 51d	400
51 to 76d	200
76 to 121d	150
121 to 201d	100
201d and coarser	50

The skein testing is done on a machine having capacities of 150 pounds and 300 pounds at a speed of 12 ± ½ inches per minute. Instead of jaws, spools which are at least 1 inch in diameter and at least 1 inch wide, of which at least one spool shall be free to turn on its axis, are used. The skein is slipped over the spools, opened out into a band, and the slack is held by the hand until the machine tightens it and finally breaks the skein.

In the single-strand test, the sample is drawn off from the side of the package. If the yarn is cut into convenient lengths, the ends should be knotted to prevent loss of twist. The jaws of the machine may be flat grip, capstan, drum, or any other device which does not have more than 25% of the specimens breaking within ½ inch of the jaw; specimens which break within ½ inch of the jaw are discarded. The speed of the lower jaw is 12 ± ½ inches per minute, and the gage length is 10 ± $\frac{1}{16}$ inches except in the A.S.T.M. test for rayon which uses a gage length of 18 inches. A great many tests are necessary for a good average. The A.S.T.M. specifies fifty tests which are free from breaks at the jaw. If the stretch is to be determined, the specimen is put into the machine with a standard initial tension applied by hanging a small weight on the specimen.

The A.S.T.M. test for tire cords specifies single-strand tests (at least six tests) on a machine with a capacity of 50 pounds with cam-clamp jaws and spool having a diameter of not less than ½ inch, and an initial tension of 4 ounces. Otherwise, tire cords are tested as in any single-strand test.

The serigraph test is so called because of its early association with the silk industry. Haven's method is the simplest and requires no special apparatus. A skein is wound as for the skein test, the yarn is spread out on the arms of the reel and stickers are pasted across the yarn above and below it and 3 inches apart; this sample is cut out of the skein and tested in a strength machine like a cloth strip sample.

Each method of testing has its advantages and disadvantages. The skein test was the earliest one used and so is more popular, and a considerable amount of data is available for comparison when this test is used. It will grade a series of yarns in the same relative order as the other tests. A fabric tester may be used by replacing the jaws with spools. In this test, as soon as a few yarns break, the rest of the skein slips, so that the test tends to give low results and not the true breaking strength. The reeling of the skein also affects the result. Unequal tension on different circuits of the yarn will give low results by breaking the shortest circuit first and then allowing slippage. In the case of rayon and silk, the skein flies apart as soon as breaking starts, and the skein test cannot be successfully used on these fibers. Then, too, yarns are not used in the form of skeins; in actual use, the load comes on the individual yarns. The skein test does not give any idea as to the uniformity of the yarn strength. It does not allow for stretch measurements, and it cannot be used for comparing yarns of different fibers.

The single-strand test more nearly represents the actual loading of the yarns in weaving. It gives a good idea as to the uniformity of the yarn and permits stretch measurements. Smaller samples may be used so that it is possible to remove short lengths of yarn from knitted or woven goods and test them. On the other hand, when this method is used, a large number of tests must be made in order to get adequate sampling and so that the average result will be accurate. A special strength-testing machine which is good for no other purpose, must be used for this test.

The serigraph test uses a cloth-testing machine and jaws. The results of this test most nearly represent the strength that the yarns will show in the cloth. This test gives the most consistent results on fine yarns. It gives a good average and adequate sampling if only one test is made to a skein, and stretch data may be determined. The serigraph test is the newest of all yarn tests and is not well known except in the silk and rayon branches of the textile industry. It combines the good points of both the skein and single strand tests.

It is sometimes desirable to compare the strength of yarns of different counts or denier. The strength of spun yarns may be converted to any specified counts by the formula:

$$N_1S_1 = N_2S_2$$

where N = counts and S = strength.

The product NS, called the strength-count product, is sometimes used, especially in the case of skein tests to compare different yarns. Rayons and silks are compared by giving the tenacity in grams per denier:

$$\text{Tenacity} = \frac{S}{D}$$

where D = denier.

If yarns composed of different fibers are to be compared by the single-strand method, the breaking length is calculated. This is the number of yards of yarn having a weight equal to the single-strand breaking strength of the yarn and is supposedly the length of yarn which would break due to its own weight.

$$\text{Breaking length of spun yarns} = SN \times \frac{H}{454}$$

Breaking length of rayons and silks

$$= \frac{S}{D} \times 15.4 \times 638 = \frac{S}{D} \times 9{,}850$$

where

S = single-strand breaking strength in grams
N = counts
H = number of yards in standard hank
D = denier

It will be noted that the formulae for breaking length contain the quantities SN and S/D which are methods of comparison already given.

TESTING FIBERS

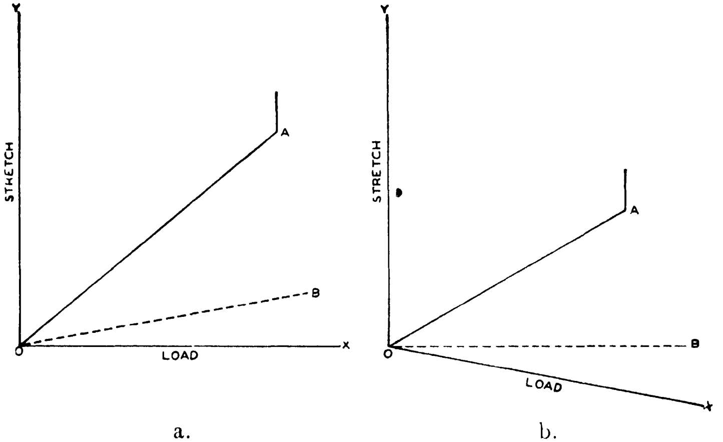
Fibers may be tested either singly or in bundles. Individual fiber testers have already been described and classified; these tests are slow and laborious and several hundred tests are necessary for a satisfactory average. Bundle tests may be carried out by the Walen method, Grimes method, or the Chandler method (A.S.T.M. standard method). The Walen method consists of parallelizing the fibers by pulling as for staple, brushing out all fibers less than $\frac{7}{8}$ inch long, cutting the bundles to $\frac{7}{8}$ length, and adjusting the bundles to weight about 0.004 grams each. The ends of the bundles are cemented together with collodion, and the bundles are broken in a ten-pound tester with a gage length of $\frac{1}{2}$ inch. Five or more bundles are tested, and the results are calculated to strength in ounces of fiber equal to a unit of No. 20 yarn by the following formula:

$$\text{Strength} = \frac{\text{sum of strength in pounds}}{\text{sum of weights in grams}} \times \frac{16 \times 454 \times 7}{840 \times 20 \times 36 \times 8}$$

The Grimes method is similar to the Walen in that the fibers are combed out and cut to a definite length. The bundles are then adjusted to a weight of 0.025 ± 0.005 grams, the ends are taped together with drafting tape, and the bundles are broken in an ordinary fabric-testing machine. The results are calculated to pounds strength per gram of fiber and are averaged. In the Chandler bundle method, a bundle of fibers is combed out parallel and the bundle is wrapped with No. 20 sewing thread on a special apparatus. The length of thread for ten revolutions is measured and the circumference of the bundle is calculated and corrected for the thickness of the thread. The samples are broken in a machine having a capacity of 300 pounds with special jaws, and each break is corrected to a standard circumference of 0.125 inches. Each corrected break is then calculated to pounds per square inch by dividing by the cross-sectional area (0.0012434 square inches), the average of ten breaks is taken.

Stretch Determination

The stretch of the textile specimen is most accurately determined by an autographic recorder mechanism as described previously. This consists of a pen which is moved horizontally by attachment to the pendulum and a platform holding the paper moved vertically by the lower jaw of the strength machine. A little consideration will show, however, that the vertical motion of the lower jaw is not only the stretch of the specimen but also includes the motion of the upper jaw, since the upper jaw moves downward a small distance for any motion of the pendulum. In a, Fig. 37, the line OA represents the autographic record of a break on a cotton yarn but ordinates of this yarn are not stretch alone but also include the motion of the upper jaw. If a piece of rigid material such as steel is clamped between the jaws, so that the motions of the upper and lower jaws are the same, the line OB will be drawn which gives us the motion of the upper jaw for various loads. The true stretch of the cotton yarn, then, is the difference between the ordinates of OA and OB. In order to correct for this automatically, modern stretch recorders are made so that the pen moves on a downward sloping guide at the same angle downward as the angle BOX. The same cotton yarn run in this manner is shown in b, Fig. 37 in which the horizontal line is now the motion of the upper jaw. The ordinates of the line OA (above the horizontal)



a. b.

FIG. 37. Stretch-Load Diagrams, Motion of Upper Jaw

now represent stretch alone. On stretch-load diagrams following this, it is assumed that this correction is made and that the ordinates represent stretch value.

Where total stretch is the only quantity desired, it may be obtained by fastening a spring tape to the bottom jaw of the machine and having it unroll past an index on the top jaw. A snap reading of the tape is taken just as the sample breaks and this gives us total stretch or *stretch at the break*.

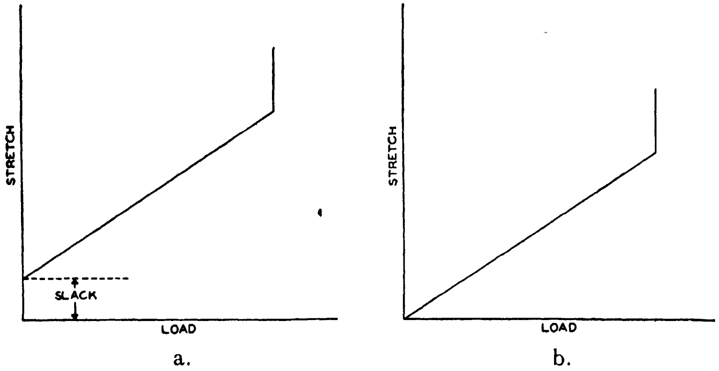


FIG. 38. Stretch-Load Diagrams, Slack of Specimen

Stretch charts, however, are preferable because they give more information about the sample. Stretch charts will tell us:

1. The total stretch of the sample
2. The manner of breaking, whether abrupt or prolonged
3. The elastic limit, if any
4. The area under the curve may be measured. This gives us the work necessary to break the sample, which in turn is a measure of the toughness of the sample.

If any sample is put into the jaws of the machine loosely, there will be a certain initial motion of the lower jaws, when the slack of the specimen is taken up before any load is applied to the sample; this reading should be deducted from any stretch readings (a, Fig. 38). If the sample is placed in the jaws under a small initial load which is enough to straighten but not to stretch the sample, this initial period is absent and the diagram gives stretch directly (b, Fig. 38). The initial tension in the case of fabrics is specified by the A.S.T.M. as 6 ounces. Diagrams given after this will assume that this slack has been corrected for or eliminated by this initial tension.

Figure 39 gives typical but somewhat idealized stretch-load diagrams of the different types of yarns, idealized only in the sense that small fluctuations have been smoothed out on the curve. That yarns made up of the natural vegetable fibers give a stretch which is directly proportional to the load applied up to the breaking point is shown in a, Fig. 39 (A, B, C). If we take the case of cotton, the breaking strength is OI and the total stretch is OH. The slope of the curve (OH/OI) is the stretch per unit load and has been found to be dependent principally on the material in the yarn, and treatments which change this material change the slope. Thus, it is possible to use this slope to determine whether a given chemical treatment which might not have affected the strength has altered the material.

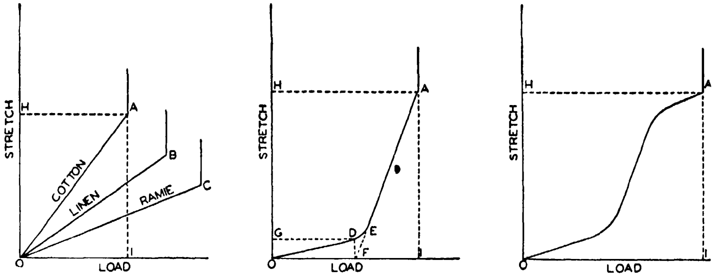


FIG. 39. Typical Stretch-Load Diagrams of Yarns

a. Vegetable fibers

b. Rayons and silks

c. Woolens and worsteds

Rayons and silks show a different type of diagram (b, Fig. 39). In this diagram, we have first a portion OD which is a straight line representing the elastic stretch; at D, elongation or plastic flow begins; at point E, the elastic stretch is completed and only elongation is left up to the point A where the yarn breaks. If the line AE is continued to the point of zero stretch (F), we have the load (OF) at which the yarn would begin to flow if it had no elasticity; this load (the yield point) should theoretically be the same load as that at the point D where the elongation begins (GD). Usually, this is true, but sometimes the two loads are not exactly the same. To sum up, the following information may be determined from this diagram:

Loads (usually expressed in grams per denier)

Breaking strength = OI
Yield point = OF
Elastic limit = GD
These last two should be equal.

Stretch measurements (usually expressed as per cent of the gage length)

Total stretch = OH
Elasticity = OG
Elongation = GH

Wet rayon does not give this type of curve but gives a single straight line with a steep slope.

A typical curve for a woolen or worsted yarn is shown in c, Fig. 39. This type of yarn has been less thoroughly studied so we cannot draw as much information from it. It will be noted, however, that this diagram starts off similar to the one for rayons and silks but that above a certain load a further change in the curve appears. This second change may be due to a change in the molecular structure of the wool. In the case of all the yarns, the area between the curve OA and the load axis gives a measure of the work done in breaking the sample and hence the toughness of the yarn.

A cloth made of a certain type of yarn shows the stretch diagram of that yarn with the addition of an initial curved portion which represents the straightening of the vertical yarns due to removal of crimp. Figure 40 shows a stretch-load diagram of cotton cloth in which we have the straight line of cotton yarn superimposed on this initial curve. In this curve,

Breaking strength = OI
Total stretch = OH
Crimp = OJ

This diagram gives us, then, a rather rough determination of crimp in addition to the strength and stretch data. This method of determining crimp is not as accurate as the method described in Chapter 5 because of the small size of the curve from which the measurements must be taken. As in the case of yarn, the area under the curve gives us the work required to break the sample. The slope of the straight line portion of the curve (JH/OI) gives us information as to changes in the material of the fabric. Rayon and woolen cloths would give stretch-load diagrams similar to the corresponding yarns but with the crimp curve added.

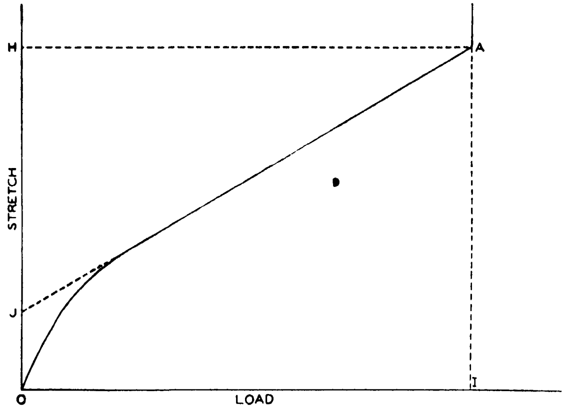


FIG. 40. Stretch-Load Diagrams of Cotton Cloth

Electric Strain Gage

This is a recently developed method of recording the stress on a sample under tension. Instead of a pendulum, this apparatus uses a cantilever arm, on the upper face of which is cemented a metal wire which changes resistance when under strain and whose change in resistance is at least approximately linear with the strain. If the upper jaw of the strength tester is attached to the end of this cantilever, the force applied results in a slight stretch of the wire with a resultant change in resistance. This change in resistance may be measured with a Wheatstone bridge. The same principle may be used for compression tests, weighings, and thickness tests. A strength tester of this sort has a negligible motion of the upper jaw and hence is a true constant-rate-of-stretch machine.

Constant Rate-of-Load Machine

We have seen in a preceding section that, on a pendulum-type strength machine, the apparent strength of a sample varies with the size of the machine, the amount of stretch of the sample, the length of the specimen, and the speed of the lower jaw. All these variables affect the final result by affecting the rate of loading of the specimen. If a constant rate-of-load machine is used, it is found that the following advantages result:

1. Rate of load on the specimen is uniform whether the specimen is long or short, rigid or stretchy, strong or weak.
2. One machine may be used for a very great range of yarns and fabrics, because the capacity of the machine has no effect on the result.
3. It makes little difference what rate of load is used; a standard rate may be used but other rates will give results which correspond within a few percent.
4. Variations in strength of a single fabric are less than on a pendulum-type machine. This means that the average result is more accurate or, on the other hand, that fewer tests may be used to get an equally accurate average.

The A.S.T.M. specifies, for rayon yarns tested by the single-strand method on a constant rate-of-load machine:

Strength *Grams per Denier*	*Rate of Loading* *Grams per Denier per Minute*
1.75 and under	2
1.75 to 3.5	4
Over 3.5	12

In the case of fabrics, the A.S.T.M. specifies a loading rate of 300 pounds per minute.

The construction of a constant rate-of-load machine is of the inclined plane type illustrated in principle in Fig. 41. As the plane is tiled downward on the right side, the rolling carriage puts more and more load on the specimen in the clamps. In order to get the constant rate-of-load, the tilting is done by a screw mechanism connected with a constant speed motor (Fig. 42). Stretch may be determined from a stretch-load diagram as in the case of the pendulum machine.

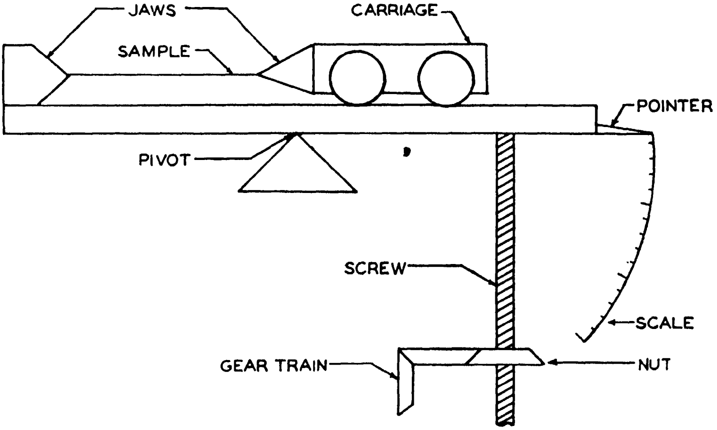


FIG. 41. Principle of Inclined-plane Strength Tester

At present, the use of the constant rate-of-load machine is not very general, principally because so many machines of the pendulum type are already in use. The advantages of this type of tester have already been given, and the disadvantages are:

1. At present, a laboratory would have to have both pendulum and inclined plane machines, since most specifications and nearly all of the accumulated data are in terms of the pendulum machine.
2. One of the most valuable uses of the strength tester is to show the variation in a sample; the inclined plane type gives less variation in results.

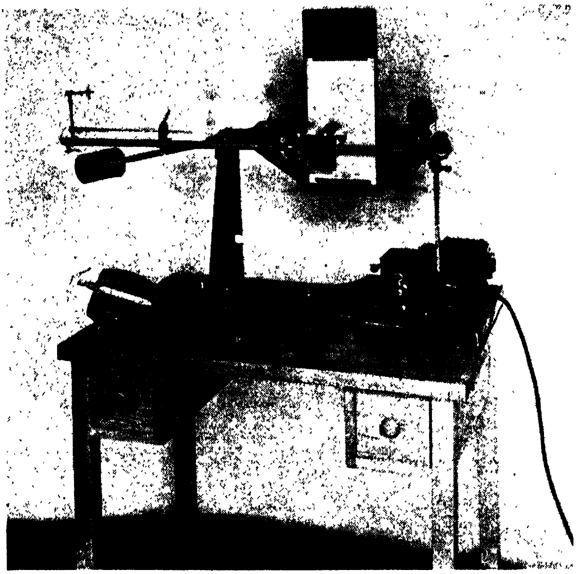


FIG. 42. Inclined-plane Strength Tester. (*Courtesy of Scott Testers, Inc.*)

3. The advantage of having one machine to test a wide range of textiles is largely offset by the fact that most plants do not use or make textile yarns or fabrics having such a wide range of strengths, so that a single pendulum machine of double range (with and without the weight on the pendulum) will take care of the whole range of any one plant's products or purchases.
4. Most testing is comparative, that is, one sample is compared with another, so that it does not really matter what type of machine is used.

Where a pendulum tester is already available, a new General Electric Company control may be added to make the machine constant rate-of-loading. It is usually better, however, to have another machine.

Ballistic Strength Machine

A strength-testing machine developed abroad and which has received little attention in this country is the ballistic or impact tester. In principle, this is a pendulum with most of its mass concentrated in the weight. The pendulum is swung from a definite height on one side of an arc and on swinging freely will swing to nearly the same angle on the other side of the arc. The specimen is attached at one end to the weight of the pendulum and, at the other end, to a fixed clamp. When the pendulum now swings, it breaks the specimen,but is slowed down so that it does not swing so far up the arc. The difference in height of the arcs swung through without a specimen and with a specimen attached is a measure of the energy used in breaking the specimen. This energy is the work of rupture obtained from the stretch-load charts of the pendulum-type strength tester.

The work of rupture is a good measure of chemical tendering because, although tendering usually decreases both strength and stretch, sometimes the strength is not affected even when the stretch is decreased. Work of rupture is directly proportional to the width of cloth broken or the number of yarns broken, so that the same size specimen is not always necessary but results on any size of specimen may be compared with results on any other size. Work of rupture is not exactly proportional to the length of specimen but becomes so by subtracting a small constant amount, so here too another variable is eliminated.

On the other hand, it has been shown that ballistic tests are more sensitive to humidity variations. It has also been shown that yarns ranked by ballistic test results show serious differences from practical results, also, as stated before, the work of rupture may be measured on a stretch-load diagram if it is desired.

Bursting Strength Machines

Bursting strength is used considerably in evaluating the strength of knit goods since strength tests are very hard to carry out. It has some use on woven fabrics which are to be stressed equally in all directions when in use, because it selects the weakest yarns (warp or filling) and breaks those, giving then the lowest pressure the cloth will withstand. Two methods of determining bursting strength are available:

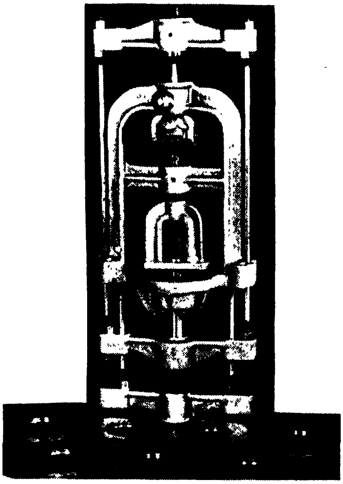


FIG. 43. Ball-burst Attachment for Strength-testing Machine. (*Courtesy of Scott Testers, Inc.*)

1. Ball burst requires special attachment for the strength tester)
2. Hydraulic burst (Mullen tester)

The ball-burst attachment for the strength tester (Fig. 43) consists of two stirrups which slide on each other. One stirrup, which replaces the top jaw, supports a post with a hard steel ball, 1 inch in diameter, held in a cup. The other stirrup, replacing the lower jaw, has two rings with internal diameters of 1.75 inches between which the specimen (3 inches square) is clamped. On starting the machine, the fabric is pulled down against the ball until bursting occurs; the strength-testing machine then indicates the bursting strength. Haven has shown that the ratio of the ball-burst strength to the strip-test strength in the weaker direction equals 4/3.

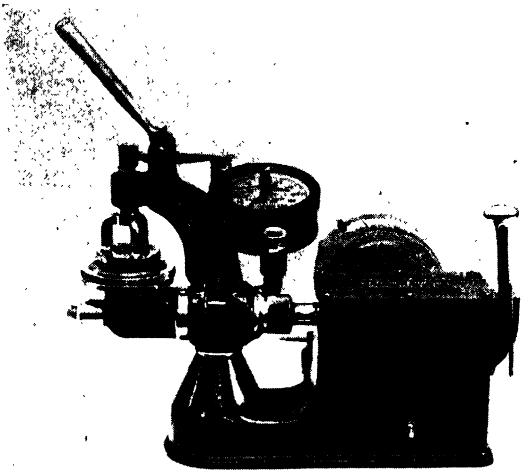


FIG. 44. Mullen Hydraulic Bursting Tester

The Mullen tester (Fig. 44) in its simplest form consists of a pump operated by a hand wheel which pumps oil or glycerin against a thin, flexible rubber diaphragm. Above the rubber diaphragm is the specimen, clamped between two rings with inside diameters of 1.2 inches. A pressure gage built on the ratchet principle records the maximum pressure when the specimen breaks. In use, the specimen is clamped in place and the hand wheel is turned at an even speed of 6 ± 0.25 cubic inches per minute until rupture occurs. The number of turns of the hand wheel and the reading of the pressure gage are then noted. This is repeated for all the specimens. To compensate for the pressure used in stretching the rubber diaphragm, the hand wheel is given the same number of turns with no specimen in place as was used with the specimen and the pressure read on the dial. This pressure subtracted from the pressure with the specimen gives the corrected bursting pressure. A newer modification of the Mullen tester has the pressure applied by a constant-speed motor and the bulge of the diaphragm and specimen record by a tilting arm and recording pen. In this case, the correction for the diaphragm is obtained from a chart of diaphragm bulge against pressure made with no specimen in place.

In theory, both methods of bursting-strength measurements suffer from the error of short lengths of fabric tested which may be less than the fiber length. In practice, this is not important since only comparisons are needed and also because it is used chiefly on knitted goods where the actual length of yarn between the clamps is two or three times the inside diameter of the rings, due to the bending of the yarn in the knitted fabric.

Effect of Moisture

The strength and stretch of all textiles are seriously affected by the regain present in the sample, which, as we have seen in Chapter 2, is in turn affected primarily by the relative humidity of the air and to a small extent by the temperature. The following tables show the effect of relative humidity on the strength

TABLE XI

RELATIVE STRENGTH AT VARIOUS RH'S

	50%	*60%*	*70%*	*80%*	*Wet*
Cotton	104	109	115	122	126
Linen	117	122	132	143	160
Jute	117	130	146	140	104
Spun viscose	97	96	92	84	57
Viscose rayon	79	74	69	62	30
Acetate rayon	90	87	82	74	60
Wool	93	90	85	81	70
Silk	90	88	86	83	65

and stretch of various yarns, on the basis of the dry strength and stretch of each yarn as 100:

TABLE XII
RELATIVE STRETCH AT VARIOUS RH'S

	50%	*60%*	*70%*	*80%*	*Wet*
Cotton	103	106	109	116	141
Linen	106	110	115	123	138
Jute	103	104	112	116	101
Spun viscose	103	106	108	110	111
Viscose rayon	123	128	135	144	194
Acetate rayon	104	106	111	120	152
Wool	144	159	175	196	310
Silk	117	119	124	129	150

As generalizations, we may say that the stretch of all textiles is increased with an increase in regain, and the strength of vegetable fibers is increased but the strength of animal fibers and the rayons is decreased by an increase in the regain. Figure

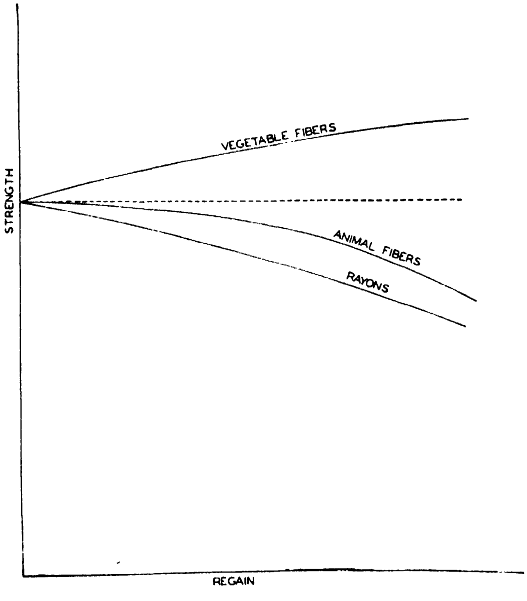


FIG. 45. Typical Strength-Regain Curves

45 shows the general relationship of strength and regain. Since the regain has such an important effect on the results and since it is impracticable to test samples when oven-dry, one of the following methods must be resorted to in order to obtain results which are comparable with results obtained by another laboratory or with results obtained at any other time by the same laboratory:

1. Test wet
2. Test at standard temperature and relative humidity
3. Test at prevailing room conditions, determine the regain, and correct the results to standard regain

TESTING WET

Testing wet was once quite common but is now done only in the case of rayons where one of the most desirable properties is a comparatively small loss in strength on wetting; rayons, there-

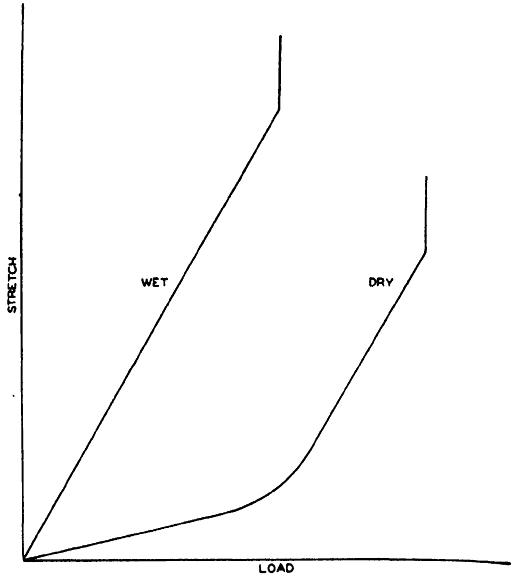


FIG. 46. Stretch-Load Diagrams for Wet and Dry Rayons

fore, are tested both wet and dry. The rayon should be soaked in tap water at room temperature for 2 hours, put in the strength tester, and tested within a minute. Results on a rayon yarn are given in grams per denier. If rayon cloth is tested (by the grab test) the number of yarns per inch in the dry state and in the wet state are counted and the wet strength is corrected to the same number of yarns as are present in the dry sample. If the stretch-load diagram of a wet rayon is obtained, it will be found that the elastic limit is missing (Fig. 46). This explains the production of rayon *shiners* found when the rayon is stretched in the wet condition, on drying, the rayon remains stretched.

TESTING AT STANDARD CONDITIONS

Testing at standard conditions of temperature and relative humidity (70° F. and 65% relative humidity) requires a special testing room (see Chapter 2) but is the most accurate, most convenient, and quickest method of testing. If the volume of testing done warrants it, this method should be used.

TESTING AT PREVAILING ROOM CONDITIONS

If a conditioning room is not available, we may test at whatever conditions prevail, obtain the amount of regain present in the broken specimens, and correct the strength to standard regain by fairly accurate formulae. The stretch of samples cannot be so corrected with any degree of accuracy. If the regain-strength curve of practically any yarn or fabric is drawn, it will be found that the middle portion of the curve, including the standard regain, is a straight line or a close approximation to a straight line, so that the equation of this straight-line portion may be used to correct the strength from one regain to another. This equation may be given as:

$$S = M \times \frac{100 + rR}{100 + rA}$$

TABLE XIII
STRENGTH CORRECTION FACTORS

Fiber Present	*Cloth Weight Ounces per Square Yard*	*Yarn Count or Denier*	*Strength Correction Factor*	*Authority*
Cotton	1½–4	above 20's	+5.2	Haven
Cotton	4–10	10's–20's	+6.0	Haven
Cotton	10–18	2's–10's	+7.7	Haven
Cotton	18–46	below 2's	+10.1	Haven
Cotton	—	all	+6.0	A.S.T.M.
Cotton tire fabric	17½	—	+6.0	Haven
Cotton tire cord	—	3/5/23's	+6.9	Haven
Cotton tire cord	—	3/5/23's	+3.5*	A.S.T.M.
Cotton fabric (two-ply)	11	—	+16.5	Haven
Mercerized cotton	3–4½	—	+3.3	Haven
Mercerized cotton	—	above 20's	0.0	Haven and others
Linen	4½	—	+8.0	Haven
Ramie	9	—	+3.8	Haven
Jute	7½–15	—	+2.5	Haven
Wool serge	6.9	—	−1.7	Haven
Camel hair	—	four-ply	−1.4	Haven
Raw silk	3.0	—	−1.1	Haven
Silk	1.9	—	−1.3	Haven
Viscose	—	100d	−2.7	Haven
Viscose	—	150d	−2.6	Haven
Viscose	—	200d	−2.9	Haven
Viscose	—	300d	−2.7	Haven
Acetate	—	100d	−3.1	Haven
Acetate	—	150d	−4.1	Haven
Acetate	—	300d	−3.8	Haven

* Correction to standard regain of 6.5%; correction applies only from 5.5 to 7% regain.

where

S = strength at standard regain
M = strength from machine reading
R = standard regain
A = actual regain
r = rate of increase or decrease of the strength for 1% change in regain, expressed as per cent of the dry strength

The value of r is independent of the form of the fabric or yarn and is dependent only on the weight of the fabric (or counts of the yarn) and the fiber of which it is composed. Values of r are given in Table XIII and standard regains have previously been referred to (Table IV).

An example of the correction method of obtaining strength is as follows:

A sample of cotton cloth of 4.25 ounces per square yard is tested for breaking strength by the grab method and gives an average of 41.5 pounds on the warp and 33.8 pounds on the filling. After breaking, a specimen is placed in a weighing bottle (tare weight of 19.5560 grams) and weighed, the weight of the bottle and sample being 21.9375 grams. On drying the sample in the oven, and weighing again, the dry weight is 21.8495 grams. What is the strength of the cloth at standard regain?

Weight of bottle and sample = 21.9375 grams
Weight of bottle and dry sample = 21.8495 grams
Weight of moisture 0.0880 gram
Weight of bottle and dry sample = 21.8495 grams
Weight of bottle alone = 19.5560 grams
Weight of dry sample 2.2935 grams

$$\text{Actual regain} = A = 100 \times \frac{0.0880}{2.2935} = 3.84\%$$

Standard regain = R = 6.5% (Table IV)
Strength correction factor = r = 6.0 (Table XIII)

Warp

$$\begin{aligned} S &= 41.5 \times \frac{100 + 6.0 \times 6.5}{100 + 6.0 \times 3.84} \\ &= 41.5 \times \frac{100 + 39.0}{100 + 23.0} \\ &= 41.5 \times \tfrac{139}{123} = 46.9 \text{ pounds} \end{aligned}$$

Filling

$$\begin{aligned} S &= 33.8 \times \frac{100 + 6.0 \times 6.5}{100 + 6.0 \times 3.84} \\ &= 33.8 \times \frac{100 + 39.0}{100 + 23.0} \\ &= 33.8 \times \tfrac{139}{123} = 38.2 \text{ pounds} \end{aligned}$$

The strength of the cloth, then, at standard regain is 46.9 pounds in the warp and 38.2 pounds in the filling.

Other Determinations with Strength Testers

FRICTION TEST

Many light and open fabrics such as gauzes, nettings, or marquisettes depend upon the friction of the yarns to hold together unless an expensive leno weave is used. To test the friction of the yarns, ravelled strip samples may be prepared as usual, then the cross threads may be ravelled down to 3 inches. At one end of the remaining cloth, alternate yarns are cut off, at the other end the other yarns are cut. The sample is inserted in the strength tester and run as a strip sample. In this case, the force to pull half the yarns longitudinally past the other half is measured.

TEARING TESTS

Two tearing tests are described by the A.S.T.M. The simplest method (tongue method) uses specimens 3 inches wide and 8 inches long, five each of warp and filling. A cut 3 inches long is made lengthwise in each specimen, starting in the center of one short edge, the pawls of the ratchet are disengaged, the specimen is placed in the machine with one tongue in the top jaws and one in the lower jaws. The force to tear is read off the stretch-load chart since it requires averaging a rather jerky load reading. The other method (trapezoid method) uses specimens 3 inches wide and 6 inches long, with the warp samples having the long dimension parallel with the filling. From these specimens, using a template, isosceles trapezoids of height 3 inches and bases 4 inches and 1 inch are cut out, and a cut, $\frac{1}{4}$ to $\frac{3}{8}$ inches long, is made in the center of the one-inch edge and perpendicular to it. Clamps, 3 inches or more in width and a gage length of 1 inch are used. The pawls are disengaged. The specimen is clamped in the machine along the nonparallel sides of the trapezoid so that the cut is halfway between the clamps, the short edge held taut and the long edge hanging in folds. The force to tear is read from the stretch-load chart as before.

SEAM SLIPPAGE DETERMINATION

The seam slippage of silk and rayon fabrics is another important determination; the determination made is the force across the seam per inch of width necessary to produce an elongation of $\frac{1}{4}$ inch in excess of the normal stretch of the fabric under the same load. A standard grab tester having a capacity of 50 pounds is used. Two specimens are cut out, one 4 × 10 inches, the other 4 × 4 inches. The smaller specimen is sewed to the longer by a standard seam $\frac{1}{2}$ inch from the end. A needle approximately 0.030 inches in diameter, No. 00 white mercerized cotton thread, 14 stitches to the inch, and a plain stitch should be used. A stretch-load chart is made on the long strip; then, using the same chart, on the seam. With a pair of dividers, the difference in the two curves at a force of one pound is measured, the dividers are set for this distance plus $\frac{1}{4}$ inch and run along the curves until the force where the two curves are separated by this distance is found. This force minus the 1 pound is the resistance to slippage of the seam.

DETERMINATIONS ON FELTS

Felts may have two determinations made on them, breaking strength and splitting resistance. For breaking strength, specimens 2 inches wide and 10 inches long are used, jaws are 2 inches wide, gage length is 3 inches, jaw speed is 12 ± ½ inches per minute. Results are given in pounds per two-inch width. For splitting resistance, the same machine, jaws, and specimen sizes are used. The specimen is split in thickness by a knife cut in the middle third of the thickness for 2 inches from one end, one lip is clamped in each jaw, and the force to pull the two sections wholly apart is measured. The splitting resistance is not determined on felts less than $\frac{3}{16}$ inch thick.

Repeated Stress Tests

As we have seen early in this chapter, textile materials are not ordinarily subjected to a single large load in actual use, but rather to a large number of fluctuations in load or a number of repeated small stresses. Tire cords, for example, are continually being given stresses much smaller than their breaking strength, and finally, they fail due to the accumulated effects of these numerous small stresses rather than to a single large one. It would be much more like actual conditions of use, then, to test the strength in this manner, and of late years much more attention has been given to tests of this sort. Some strength-testing machines are now so built as to make it possible to automatically apply and remove a definite load for an infinite number of times.

The most significant way to measure this *fatigue* of textiles would be to find that load which the specimen would bear indefinitely or for a fixed large number of applications, but this would be a slow and laborious task. Peirce has suggested the measurement of the number of applications of a load equal to 60% of the break-load which would cause rupture of the specimen. Figure 47 shows the first two cycles of load applied to a cotton yarn where the load is brought to a certain value each time. Further cycles do not differ very much from the second cycle, but it will be noted that the second cycle is distinctly different from the first.

At present, tests of this nature are still in the research stage and no standards of method or results have been set up as yet.

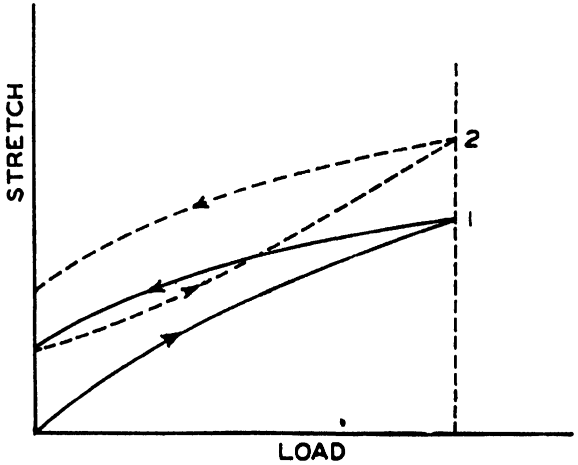


FIG. 47. Stretch-Load Diagram of Cotton Yarn with Repeated Stress Applications

Accelerated Aging Tests

In order to determine quickly the effects of aging on a textile sample, both the A.A.T.C.C. and the A.S.T.M. have developed accelerated aging tests. Each of these depends upon measuring the strength of the material before and after the test and expressing the results as a percentage of the original strength.

The A.A.T.C.C. method involves heating the fabric for 6 hours in an oven at 135° F ± 2°, using 25 grams of sample for each cubic foot of oven capacity. At the beginning of the second, third, fourth, fifth, and sixth hours all ports are closed and 20 cubic centimeters of water are added for each cubic foot of oven capacity. The ports are opened again after 5 minutes.

The A S.T.M. method uses the Fade-Ometer (a glass-enclosed carbon arc) which is ordinarily used for light-fastness tests on dyed fabrics. The samples to be tested for strength are exposed in this machine for 20 hours.

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PART II

CHEMICAL TESTING

Chapter 11

INTRODUCTION AND GENERAL CONSIDERATIONS

Before discussing individual chemical tests, it is necessary to consider some of the generalizations which apply to all chemical tests. In the first place, it will be observed as the tests are taken up that there are two general types of determinations—direct and indirect. In a direct determination the constituent is determined by a direct weighing or titration; in an indirect determination, the constituent is determined by the loss in weight of the sample or by a change in some property of the sample. It is generally considered better to make a direct determination if it is at all possible, although if carefully done, an indirect determination may be as accurate or sometimes more accurate.

All chemical tests on textile materials are based on a dry weight of sample. Therefore, the sample should be oven-dried and weighed before the test is carried out. If this is not possible, because of chemical changes which occur on heating, two samples are weighed out, *at the same time;* on one of these samples the chemical test is carried out and on the other a regain determination is made. The dry weight of the sample used for the chemical test is then calculated from the results of the regain determination. The formulae have already been given but will be reviewed here. Let

A = weight of weighing bottle + sample to be dried
B = weight of weighing bottle + dry sample
C = weight of weighing bottle
R = regain in per cent
W = weight of sample weighed out for the chemical test
D = dry weight of sample for the chemical test

Then,

$$R = \frac{A - B}{B - C} \times 100$$

and

$$D = \frac{W}{1 + \frac{R}{100}}$$

As an example, let us consider a copper number determination in which the following data were obtained:

Weight of bottle and sample to be dried = 23.9765 grams
Weight of bottle and dry sample = 23.8785 grams
Weight of bottle alone = 21.6830 grams
Weight of sample for test = 2.59 grams

$$R = \frac{23.9765 - 23.8785}{23.8785 - 21.6830} \times 100 = \frac{0.0980}{2.1955} \times 100 = 4.47\%$$

$$D = \frac{2.59}{1 + \frac{4.47}{100}} = \frac{2.59}{1.0447} = 2.48 \text{ grams}$$

Although determinations are generally made in the laboratory on the dry weight of the sample and results are expressed in terms of a dry basis, the non-chemist may sometimes want the results in terms of the *normal* material, by which is meant in terms of a sample with its standard regain. This may be figured easily from the dry-basis results. Two cases are possible which will be illustrated by concrete examples.

A sample of cotton cloth contains 2.56% ash (dry basis), what is the per cent ash at standard regain? Here we have 2.56 parts of ash which do not pick up any regain together with 100.00 − 2.56 = 97.44 parts of cotton cloth which pick up 6.5% regain.

At standard regain,

$$\begin{array}{rrl} 1.065 \times 97.44 = & 103.85 & \text{parts of cotton} \\ + & \underline{2.56} & \text{parts of ash} \\ & 106.41 & \text{total parts} \end{array}$$

$$\text{Ash} = \frac{2.56}{106.41} \times 100 = 2.40\%$$

A sample of cloth containing silk and cotton, on analysis, shows 21.5% silk and 78.5% cotton (dry basis). What are the amounts of silk and cotton at standard regain? Here we have two substances, both of which pick up regain but in different amounts.

At standard regain,

$$\begin{aligned} 1.065 \times 78.5 &= 83.7 \text{ parts of cotton} \\ 1.11 \times 21.5 &= \underline{23.9} \text{ parts of silk} \\ & 107.6 \text{ total parts} \end{aligned}$$

$$\text{Cotton} = \frac{83.7}{107.6} \times 100 = 77.8\%$$

$$\text{Silk} = \frac{23.9}{107.6} \times 100 = 22.2\%$$

Since it is impossible to do much chemical textile testing without previous training in chemistry, it is assumed throughout this section that the reader is familiar with the usual qualitative tests and also with the technique of quantitative analysis. Where the expression ***run a blank*** is used, it means that the procedure (usually a titration) should be carried out on the chemicals used without any sample present. The results may then be used to correct a direct determination for impurities present in the reagents or to furnish necessary data for an indirect determination. Unless otherwise indicated, temperatures are given in degrees Centigrade; weights in grams; and volumes in cubic centimeters (or milliliters).

In addition to the usual classification of tests as qualitative or quantitative, we may further classify chemical textile tests into the following three main groups:

1. Those for extraneous matter present, both inorganic and organic
2. Those for fibers present
3. Those for condition or state of the fibrous material

Chapter 12

GENERAL AND INORGANIC EXTRANEOUS MATTER

General

The extraneous matter, both inorganic and organic, which may be added to a textile material during manufacturing consists of one or more of the following substances listed under the processes or treatments in which they are used:

- Processing
  - Acids
  - Alkalis
  - Soaps
  - Lubricants
- Dyeing
  - Mordants
  - Dyestuffs
  - After treatments to improve fastness
- Altering the handle or appearance
  - Lubricants
  - Softeners
  - Deliquescents
  - Delusterants
  - Weighting materials
  - Adhesives and stiffening agents
    - Natural
    - Cellulose derivatives
    - Synthetic resins
- Improving the use characteristics
  - Antiseptics
  - Waterproofing
  - Flameproofing

Mothproofing
Slipproofing
Shrinkproofing
Creaseproofing or resiliency improvement

The substances now being used for each of these purposes are listed in Table XIV. They are divided into the commonly used substances and the less frequently used substances.

DYEING

In the field of dyeing, acid or mordant dyes are most frequently used on wool except when solid shades are desired in a mixture with cotton or rayon. If a mordant dye is used, the most common mordant is chrome, but occasionally aluminum, iron, antimony, tin, or copper mordants are used. In dyeing viscose or cotton, the dyes most frequently used are direct cotton, vat, sulfur, or naphthol colors. For acetate and nylon, the acetate-type dyes are principally used. Basic dyes are seldom used, mineral dyes are confined principally to tents and awnings, and metallized dyes are not yet classed as commonly used dyes. One class of wool dyes is frequently afterchromed to improve its fastness. Some direct cotton dyes may be aftertreated with copper sulfate or formaldehyde for the same purpose. Certain cationic finishes have been found to improve the fastness of dyes to crocking, and synthetic resins often improve dye fastness to washing and sometimes to light.

ALTERING HANDLE OR APPEARANCE

Lubricants are usually oils in the case of wool, or waxes in the case of cotton; rosin and tallow are now less frequently used. Softeners may be soaps, sulfonated oils, sulfated alcohols, or the newer cationic softeners which have a direct affinity for the fibers and, therefore, are permanent; glycerin and glycols are still used to some extent, and the use of stearamide is increasing. Glycerin and glycol are also the most commonly used deliquescents; magnesium chloride and calcium chloride

TABLE XIV

EXTRANEOUS MATTER IN TEXTILES

	Common Materials		*Uncommon Materials*	
	In Textiles	*In Ash*	*In Textiles*	*In Ash*
Mordants	Chromium oxide	Cr_2O_3	Oxides of Al, Fe, Sb, Sn, Cu	Al_2O_3, Fe_2O_3, Sb_2O_3 SnO_2, CuO
Dyes	Acid, mordant, direct, vat, sulfur, naphthol, acetate	Na_2SO_4, Na_2CO_3	Basic, metalized, mineral	NaCl, CuO, Cr_2O_3, MnO_2, Fe_2O_3, $PbCrO_4$
Aftertreatments	Chromium oxide	Cr_2O_3	Cationic finish, formaldehyde, copper sulfate, synthetic resins	NaCl, $CuSO_4$
Lubricants	Oils, waxes		Rosin, tallow	
Softeners	Soaps, sulfonated oils, sulfated alcohols, cationic softeners	Na_2CO_3, Na_2SO_4, NaCl	Glycerin, glycol, stearamide	
Deliquescents	Glycerin, glycol		$MgCl_2$, $CaCl_2$	$MgCl_2$, $CaCl_2$
Delusterants	TiO_2, $BaSO_4$, clay	TiO_2, $BaSO_4$, SiO_2, Al_2O_3		
Weighting	SnO_2, SiO_2, clay	SnO_2, SiO_2, Al_2O_3	Al_2O_3, P_2O_5, $MgSO_4$, Na_2SO_4, iron tannate, Prussian blue, sugars	Al_2O_3, P_2O_5, $MgSO_4$, Na_2SO_4, Fe_2O_3

Natural adhesives	Starch, dextrin		Gums, mosses, albumen, casein, glue, gelatin	CaO
Cellulose derivatives	Ethyl cellulose		Methyl cellulose, colloidally activated cellulose	ZnO
Synthetic resins	Urea-formaldehyde, melamine-formaldehyde		Phenol-formaldehyde, vinyl, styrene, acrylate, alkyd	
Antiseptics	$ZnCl_2$, Cu and Zn naphthenates, phenols, cresols	$ZnCl_2$, CuO, ZnO	Na_2SiF_6, H_3BO_3, $CuSO_4$, copper soaps, salicylic acid, turpentine, organic mercury compounds	Na_2SiF_6, H_3BO_3, $CuSO_4$, CuO
Waterproofing	Al_2O_3, wax, pyridine derivatives	Al_2O_3	Vinyl chloride, other synthetic resins	
Flameproofing	Chlorinated paraffin, ammonium sulfamate, Sb_2O_3, $CaCO_3$	Sb_2O_3, $CaCO_3$	Ammonium salts, alum, Na_2SnO_3, Na_2SiO_3, Na_2HPO_4, $Na_2B_4O_7$, H_3BO_3	$Al_2(SO_4)_3$, Na_2SnO_3, Na_2SiO_3, Na_2HPO_4, $Na_2B_4O_7$, H_3BO_3
Mothproofing	NaF, CaF_2, AlF_3	NaF, CaF_2, AlF_3	Alkaloids, organic fluorides	NaF
Slipproofing	Synthetic resins, cellulose derivatives		SiO_2	SiO_2
Shrinkproofing	Synthetic resins			
Creaseproofing	Synthetic resins			

are becoming much less common. Titanium dioxide is used by the rayon manufacturers to deluster rayon, but, in the finishing plant, barium sulfate or sometimes a clay, which is colloidally active and gelatinous in nature, is used. Weighting of cotton goods is still done principally with China clay. Silk is weighted usually by the tin-silicate method, although sometimes it may be weighted by aluminum instead of tin, or phosphate in place of silicate. Silk is still weighted by iron tannate and Prussian blue, and occasionally by sodium sulfate, magnesium sulfate, or sugars.

Starch and dextrin are still the most commonly used natural stiffening agents, but gums, mosses, albumen, casein, glue, or gelatin may be used along with them to modify their properties. Ethyl cellulose has been used frequently where a permanent finish is desired, since it is not removed in laundering. Methyl cellulose is water soluble but is sometimes used in place of starch. Colloidally activated cellulose (C.A.C.) consists of a solution of cellulose in sodium hydroxide using a zinc catalyst; it gives a permanent finish when deposited on a fabric.

Improving Use Characteristics

The most commonly used antiseptic for cotton and rayon goods is still zinc chloride, but copper or zinc naphthenate is used now much more generally than before the second world war. Phenols and cresols, as well as their salts, are also more frequently used. The older silico-fluorides, boric acid, copper sulfate, salicylic acid, and turpentine mildew antiseptics are still occasionally encountered. The use of various organic mercury compounds seems to be increasing.

The most common waterproofing is still aluminum acetate, either alone or in combination with a wax emulsion. The use of various pyridine or piperidine condensation products, such as Zelan, is also common. Vinyl chloride is used in such materials as shower curtains, bathroom drapes, and transparent raincoats. Occasionally, other synthetic resins are also used to give some water resistance.

Several flameproofing or flame-resistant finishes are also available. Ammonium sulfamate is one material used. Chlorinated paraffins, either alone or in combination with antimony oxide or calcium carbonate, were used to a great extent in the last war. Other agents sometimes used are ammonium salts, alum, sodium stannate, sodium silicate, sodium phosphate, and borax or borax and boric acid mixtures.

Wool and mohair fabrics are mothproofed principally by mixtures of inorganic fluorides, but occasionally alkaloids or organic complex fluorides are used.

One drawback to open rayon fabrics has always been the ease with which the rayon yarns slip out of place and bunch up together. This is largely prevented by slipproofing, usually with synthetic resins but sometimes with colloidal silica. Synthetic resins are also used to control the shrinkage of cotton, rayon, and even wool fabrics as well as to give resiliency or creaseproofing to vegetable fibers and rayons or to improve the resiliency of wools.

Mineral Matter

ASHING

It is usually necessary to determine the amount and often the qualitative composition of all of the mineral matter present in a textile sample. To do this, an ash determination must be made on the sample. Two procedures may be used, depending on whether a burner or a muffle is used. A sample is dried and weighed and is then placed in a tared porcelain or silica crucible. If a burner is used, the sample is ignited gently until it is all carbonized, i.e., until no more fumes come off, and is then ignited strongly until all the carbon is burned off. The crucible and contents are cooled in a desiccator and weighed. When two weighings, with an ignition lasting for half an hour between them, are substantially identical, the ashing is complete. If a muffle furnace is available, the crucible containing the sample is placed in front of the open muffle until the carbonization is carried out and the ignition is completed inside the muffle at a temperature of about 800°C. If we let

a = weight of dry sample
b = weight of empty crucible
c = weight of crucible and ash

then,

$$\text{Per cent ash} = \frac{c - b}{a} \times 100$$

The above determination may be in error because:

Carbon dioxide may be lost from carbonates.

Water may be lost from inorganic matter containing water of crystallization or from hydroxides.

Ammonium salts may be volatilized and lost.

A few inorganic materials may sublime. These are comparatively few and unlikely to be present in textiles.

SOURCES OF THE MINERAL MATTER

The mineral matter present in textiles may be derived from one or more of the following sources:

The natural ash of the fiber
Extraneous matter added during manufacture
Accidental spots and stains

Natural Ash

The natural ash content of textile fibers is given in Table XV. It will be observed that the ash content of the raw fibers varies considerably but that scouring or bleaching not only decreases the amount of ash but also makes it more nearly constant from one sample to another. It is usually possible to decide from an ash determination and Table XV whether there is any additional mineral matter present. Thus, a sample of scoured and dyed cotton with an ash content of 0.65% will almost certainly contain mineral matter in addition to its average natural ash content of 0.25%. If additional mineral matter is found to be present, the ash should be analyzed qualitatively for the constituents. The last part of Table XV gives the qualitative analysis of the natural ash of cotton, wool, and silk so that allowance may be made for these constituents in considering the results of the qualitative tests on the ash.

TABLE XV
NATURAL ASH CONTENT OF FIBERS

Fiber	*Usual Range* %	*Average* %	*Maximum* %
Cotton (raw and mechanically clean	0.9–1.2	1.0	1.3
Cotton (scoured)	0.2–0.3	0.25	
Cotton (bleached)			0.1
Ramie (raw)	2–6		6.0
Ramie (purified)		1.75	
Flax	1–6		6.0
Jute	0.9–2.0		
Rayons (not delustered)	0.1–0.5	0.2	
Silk (raw)		1.0*	
Silk (boiled-off)		0.5	
Tussah (raw)			5.0
Tussah (purified)		0.25	
Wool (scoured)		1.0	
Wool (fine and medium)			1.0
Wool (coarse)			3.0

* A poor average, large amount of variation.

QUALITATIVE ANALYSIS OF ASH

Fiber	*Constituents Present in Large Amounts*	*Constituents Present in Small Amounts*
Cotton (raw and mechanically clean)	K, Ca, CO_3	Na, Mg, Fe + Al, SO_4, PO_4
Cotton (scoured)	K, Na, SO_4	Ca, Mg, Fe + Al, PO_4, Cl
Silk (boiled-off)	K, Na, Ca, CO_3	
Wool (scoured)	K, Ca, Fe + Al, SO_4	Na, SiO_3, CO_3

Extraneous Matter

Inorganic extraneous matter added during processing has been discussed in the first section of this chapter, and the constituents which might be found in the ash are given in

Table XIV. It will be noted that the previous division into common and uncommon materials is used again here. If a systematic qualitative analysis is run on the ash, comparison with this table will show the previous treatment of the sample. If random tests for the elements are to be made, the table will show which are the most likely to be present.

Accidental Spots and Stains

Accidental stains or spots may, of course, be of any origin. Usually, these spots are quite small so that an ordinary analysis cannot be carried out. In these cases, microchemical tests are valuable; the spectroscope may be used; or the microscope may be used to identify the constituents present. Rust stains are perhaps the most common and these may be determined by the common tests for iron.

Silk Weighting

The determination of the amount of weighting present on silk has always been of great commercial importance because of the high value of the fiber weighted. The weighting material may be: iron as Prussian blue or as iron tannate; tin as the silicate, phosphate, tungstate, or hydroxide, and occasionally zinc or lead compounds. Sometimes, logwood is applied in such large quantities as to be considered as weighting. In addition to these weighting materials, cane sugar, glucose, glycerin, gelatin, tannin, and sodium or magnesium sulfate may have been used on the silk as finishing compounds.

QUALITATIVE ANALYSIS

A qualitative analysis of the silk may be made if the processes to which it has been exposed are unknown. The identification of the organic finishing materials will be taken up in a later chapter. The weighting material may be identified by ashing a sample of the silk and testing the ash for the above-mentioned metals and acid radicals, making allowance for the decomposition of the Prussian blue (ferric ferrocyanide) and iron tannate to ferric oxide during the ignition. Tin phosphate or tin silicate weighting is by far the most extensively used at present.

QUANTITATIVE ANALYSIS

Four important methods have been suggested for the quantitative determination of weighting:

Determination of Nitrogen

The nitrogen is determined by the Kjeldahl method and, from the result, the fibroin present is calculated. This requires a preliminary removal of all dyes and any Prussian blue present, this is generally impracticable.

Determination of the Ash

This is the quickest method if tin weighting is present. However, there is some loss of water of hydration from the tin compound which makes this method somewhat inaccurate. It has been suggested that a correction factor of 1.12 be applied to the results of the ash determination to give the tin weighting.

Alternate Treatments with Sodium Carbonate and Acid

The acid used is hydrofluoric if silicate is present or hydrochloric if silicate is not present. This was a standard method at one time but has been found to be inefficient in the removal of weighting so that different analysts had difficulty in checking each other.

Treatment with a Mixture of Hydrochloric and Hydrofluoric Acids

This method has been found to be more efficient than the use of either acid singly and to give more reproducible results. The standard method of the A.A.T.C.C., which does not differ materially from the original method of Mease, will be given in detail.

a. Prepare the following solutions:

Acid reagent: 2% by weight of hydrofluoric acid and 2% by weight of hydrochloric acid in distilled water.

Sodium carbonate reagent: 2% by weight of anhydrous sodium carbonate in distilled water.

b. Take a sample of the silk, preferably across the whole width of the cloth, weighing from 3 to 5 grams. Dry at 105–110° C. for 1½ hours. Place in a tared weighing bottle. Cool in a desiccator and weigh. Let the weight of this sample = A.

c. Digest this dried sample in ninety to one hundred times its weight of distilled water at 65° C. ± 1° for 20 minutes. Squeeze out the sample. Place in forty to fifty times its weight of distilled water and heat to 65° C. Repeat this rinsing twice. Squeeze. Rinse with two portions, 30 cubic centimeters each, of warm alcohol and then rinse with two portions of ethyl ether at room temperature. Squeeze. Dry in the air. Drop the sample into a beaker of boiling water. Let it stand for 5 minutes. Squeeze, dry, and weigh as before. Let this weight = B.

d. Place the sample in ninety to one hundred times its weight of acid reagent. Heat to 55° C. ± 1°, and hold at this temperature for 20 minutes. Pour off the acid reagent. Treat with 200 cubic centimeters of distilled water at 55° C. for 5 minutes. Repeat the water treatment. Remove the sample and squeeze.

e. Repeat treatment (d).

f. Repeat treatment (d) but use sodium carbonate reagent in place of the acid reagent.

g. Repeat treatment (d) twice, using acid reagent.

h. Squeeze. Rinse with alcohol, then with ether, then with boiling water. Dry and weigh. Let this weight = C.

i. Ash the residue and determine the weight of the ash. Let this weight = D. (This treatment should give a negligible ash and step (i) is performed merely as a check on the previous treatments. It may be omitted in routine work.)

j. Calculate, using the following formulae:

$$\text{Per cent finishing materials} = \frac{A - B}{A} \times 100$$

$$\text{Per cent total weighting} = \frac{A - C + D}{A} \times 100$$

Acids and Alkalis

The problem of the determination of acid or alkali in textile goods is complicated by the fact that small quantities of acid or base are adsorbed by the fiber and held rather tenaciously. We cannot simply extract the acid or alkali from the material with water and then test the extract, even for qualitative results, nor can we suspend the material in water and titrate with the proper reagent because the reagent would be adsorbed by the fiber. For qualitative tests, we must apply the indicator directly to the fiber, and for quantitative results, we must neutralize the adsorbed acid or base with a base or acid which is not itself appreciably adsorbed and titrate the excess of the reagent.

QUALITATIVE DETERMINATIONS

On Cotton

In the case of cotton, the difficulty is not so great because much less adsorption occurs than in other materials, and the acid or base is less firmly held. Litmus paper, pressed between layers of moistened cotton goods for a few minutes, or moist litmus paper, pressed on the fabric, will give a slight color change with 0.01% acid or alkali. A more sensitive test is the use of a saturated solution of methyl red in water as an indicator. When spotted directly on the fiber, this turns red with 0.005% acid and yellow with 0.005% alkali.

On Wool

There is no satisfactory qualitative test for acids in wool which will work on traces of acid but Clayton's methyl green test is available for traces of alkali. A sample of the wool weighing 0.25 gram is boiled in 200 cubic centimeters of water containing 1 cubic centimeter of a 0.1% solution of methyl green (Colour Index No. 684) in a white silica dish. If alkali is present to the extent of 0.04% in the wool, complete decolorization will result.

QUANTITATIVE DETERMINATIONS

In the quantitative determination, regain is determined on a separate sample, as described in the introduction to this section, while the chemical test is being carried out on the other samples.

On Cotton

The method using N/50 sodium hydroxide, which is accurate to 0.02% acid or alkali, is satisfactory on cotton goods. It is carried out as follows:

Boil 100 cubic centimeters of distilled water in a conical flask. Add 1 cubic centimeter of 0.5% phenolphthalein and then N/50 sodium hydroxide until a very faint pink color is obtained against a white background. Add a three-gram sample of the cotton goods to be tested and boil for a few minutes. If the pink color disappears, titrate with N/50 sodium hydroxide until the same pink color is obtained again. The blank on a perfectly neutral cotton fabric is equal to 0.10 cubic centimeter of N/50 sodium hydroxide. If the addition of the sample causes a deepening of the color instead of discharging it, add an excess of N/50 sulfuric acid to discharge the color and then titrate with N/50 sodium hydroxide as before. A sample set of data and calculations is given below:

Determination of Sulfuric Acid in Cotton

a. Weight of bottle and sample	44.3450 grams
b. Weight of bottle and dry sample	44.2535 grams
c. Weight of bottle alone	40.7546 grams
d. Weight of moisture (a − b)	0.0915 grams
e. Weight of dry sample (b − c)	3.4989 grams
f. Regain $\left(\frac{100\,d}{e}\right)$	2.62%

g. Weight of sample for test	3.2429 grams	3.1077 grams
h. Dry weight of sample $\left(\frac{g}{1 + \frac{f}{100}}\right)$	3.1962 grams	3.0284 grams
sample S1 + 100S	3.1962 grams	3.0284 grams
i. Volume of sodium hydroxide	2.10 cubic centimeters	1.60 cubic centimeters
j. Blank on neutral fabric	0.10 cubic centimeter	0.10 cubic centimeter
k. Corrected volume of sodium hydroxide (i − j)	2.00 cubic centimeters	1.50 cubic centimeters
m. Normality of sodium hydroxide	0.0197	0.0197
n. Milliequivalent weight of sulfuric acid	0.049	0.049
o. Sulfuric acid $\left(100k \times m \times \frac{n}{h}\right)$	0.062%	0.064%
p. Sulfuric acid (average)		0.063%

On Wool

Tests for Acid

For the determination of acid in wool, several methods are available all of which may be classified under the following four general methods:

1. Neutralize the acid with a weak base which is itself not adsorbed and titrate the excess of the base.

   a. Ammonia method
   b. Sodium bicarbonate method
   c. Triethanolamine method

2. Treat with a soluble salt which will liberate an insoluble acid. Remove the acid and titrate the excess of the salt.

    a. Sodium terephthalate method

3. Treat with a soluble salt which will liberate a volatile acid. Distill over the acid and titrate it.

    a. Sodium acetate method

4. Extract the acid with a base and titrate it directly in the base using phenolphthalein as the indicator.

    a. Pyridine method

The ammonia and sodium bicarbonate methods are less satisfactory than the triethanolamine method. The sodium terephthalate method has not proved satisfactory. The sodium acetate method is slow and tedious but gives satisfactory results and is especially valuable when the wool is dyed with a color which bleeds easily. The triethanolamine method and the pyridine method are recommended for most cases.

The triethanolamine method is described here and a sample calculation is shown.

Soak samples weighing 2.5 grams each overnight in 50 cubic centimeters of N/10 triethanolamine and 50 cubic centimeters of water. Filter. Wash with four washings of 75 cubic centimeters each of hot water. Titrate the combined filtrate and washings with standard N/10 acid, using methyl red indicator. Run a blank on 50 cubic centimeters of triethanolamine and the same amount of water as was used for washing above.

Determination of Sulfuric Acid in Wool

a. Weight of bottle and sample	51.1408 grams
b. Weight of bottle and dry sample	50.9646 grams
c. Weight of bottle alone	48.6136 grams
d. Weight of moisture (a − b)	0.1762 grams

e. Weight of dry sample (b − c)		2.3510 grams
f. Regain $\left(100\frac{d}{e}\right)$		7.47%
g. Weight of sample for test	2.7680 grams	2.5330 grams
h. Dry weight of sample $\left(\frac{g}{1+\frac{f}{100}}\right)$	2.576 grams	2.357 grams
i. Volume of acid for blank	59.67 cubic centimeters	59.67 cubic centimeters
j. Volume of acid for sample	55.58 cubic centimeters	56.04 cubic centimeters
k. Volume of acid corresponding to acid present in the wool (i − j)	4.09	3.63
m. Normality of acid used	0.0926	0.0926
n. Milliequivalent weight of sulfuric acid	0.049	0.049
o. Sulfuric acid $\left(100 \times m \times \frac{n}{b}\right)$	0.72%	0.70%
p. Sulfuric acid (average)		0.71%

The sodium acetate method is carried out as follows: Place a two-gram sample of wool and 0.5 to 1.0 grams of sodium acetate in a distilling flask having a capacity of 500 cubic centimeters with water and steam distill until 50 cubic centimeters have distilled over. Titrate the distillate with N/10 sodium hydroxide, using phenolphthalein as an indicator. This method is useful on dyed fiber where the dye is apt to bleed.

The pyridine method of Barrit as modified by Eavenson and Creely is as follows: A five-gram sample of wool is placed in a flask having a capacity of 500 cubic centimeters and 300 cubic centimeters of 0.5% pyridine solution as near the boiling point as possible are added. Shake for 5 minutes, take off an aliquot part of 150 cubic centimeters and titrate with N/20 sodium hydroxide, using phenolphthalein as an indicator. If the sample is dyed and the color bleeds, use pyridine at room temperature and aollw it to stand for an hour.

Tests for Alkali

When alkali is found in wool, soaps are included with the alkali. Two general methods are available for the determination of alkali in wool.

1. Treat with an insoluble acid which combines with the alkali to form a soluble salt, filter, and titrate the salt.

    a. Terephthalic acid method
    b. Boric acid method

2. Add a known quantity of acid to the sample, then determine the excess acid by one of the methods previously given.

The boric acid method is carried out as follows: Soak a sample of the wool in 1% boric acid for several hours. Filter, wash with water eight or ten times. Titrate the combined filtrate and washings with N/10 hydrochloric acid using methyl red or methyl orange as indicator. If dyes are present, use boric acid saturated with sodium chloride to reduce the bleeding of the dye.

The triethanolamine or pyridine methods may be applied to the determination of alkalis in wool by first adding a known amount of acid to the sample and then treating as in the determination of acid present. The difference between the acid added and the acid found will be equivalent to the alkali present.

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Chapter 13

ORGANIC EXTRANEOUS MATTER

Qualitative Analysis

For a good many years, the identification of finishing agents on textile fabrics has been a matter of trial and error. In the case of inorganic matter, it has been possible to perform an ashing or a Kjeldahl digestion, followed by a regular qualitative inorganic analysis of the resulting substance; this has been satisfactory. However, in the case of organic matter, it has been necessary to resort to trial and error, and if the first guess did not happen to be correct, further guesses and trials had to be made. This method is obviously inefficient in principle, and if several agents are present, one or more may be missed entirely.

The following suggested scheme of organic analysis is similar in principle to inorganic qualitative analysis in that the presence or absence of certain groups is determined first. Each group found to be present is then progressively analyzed for its individual components. The group reagents are solvents which dissolve certain classes of substances, and subsequent evaporation of the solvent shows the presence or absence of the group. In order to avoid time-consuming Soxhlet extractions, a simplified procedure which gives good qualitative results, is used. As a matter of fact, it is possible to obtain not only qualitative but also fairly good quantitative results by weighing the fabric after each group solvent has been used.

The antiseptics or mildewproofing agents are usually present in such small quantities that micro or semimicromethods are necessary for their detection; therefore, they have been generally omitted from this scheme of analysis. Certain other finishing agents have not been included because: (1) authentic samples could not be obtained, (2) they have come on the market too recently to be included in the investigation which resulted in this scheme, or (3) they have not yet been definitely established as commercially acceptable finishes. It is hoped that these finishes may be fitted into the scheme of analysis as soon as possible.

A little experience is necessary in judging the significance of group residues. Practically all wool samples, for instance, will leave at least a trace in groups A and B, because they contain small amounts of oil and soap from processing. These should be reported as traces so that it will be realized that they do not constitute a deliberately added finishing agent. This method of analysis is quite fast, about an hour being required for a determination when two or three components are present.

The required apparatus consists of a round-bottomed flask arranged for refluxing the liquid which is contained in a beaker, as shown in Figure 48, having a capacity of 400 cubic centimeters, several porcelain casseroles, 2½ inches in diameter, and some test tubes. It is also convenient to have a hood and electric hot plate for evaporations, but an inverted funnel connected to a filter pump and held over a ring-stand may be used in an open laboratory if desired. The chemicals and reagents to be used will be designated in the various tests. The test procedures are as follows:

Cut out discs of the fabric large enough to just cover the bottom of the 400 cubic centimeter beaker. Use enough discs to give 3 to 5 grams of sample. Place the extraction liquid, i.e., the particular solvent which is being used according to the scheme, in the beaker and place the beaker under the reflux flask.

A. Using 50 cubic centimeters of carbon tetrachloride as the solvent, heat the contents of the beaker to boiling and boil for a total time of 5 minutes. Pour the solution into a casserole. This will be called solution A. Again, add 50 cubic centimeters of the same solvent to the beaker and repeat the extraction, discarding this solution (save for recovery of solvent, if desired). Allow the sample to dry on a watch-glass before the next extraction, using an oven if one is available.

B. Using the sample from A, repeat the whole procedure using 50 cubic centimeters of 95% ethyl alcohol each time. This solution will be called solution B.

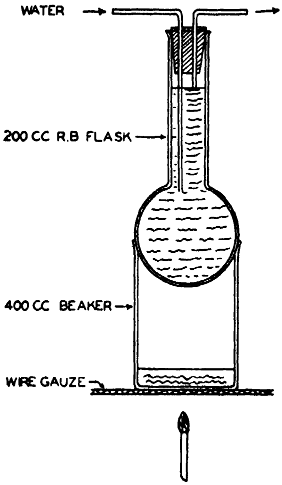

FIG. 48. Apparatus for Identification of Organic Finishing Agents

C. Using the sample from B, repeat the whole procedure using 50 cubic centimeters of distilled water each time. This solution will be called solution C.

D. Using the sample from C, repeat the whole procedure using 50 cubic centimeters of dioxan each time. This will be called solution D.

E. Using the sample from D, repeat the procedure using 50 cubic centimeters of distilled water to which 2.0 cubic centimeters of concentrated hydrochloric acid have been added. This solution will be called solution E. The second boiling may be omitted and the sample discarded after the first boiling unless fiber testing is to be done on the finish-free fabric.

There are now five separate solutions, each of which contains certain finishing materials which are to be tested for individual components. Tables XVI to XX give an outline of the tests to be performed, and the following instructions will give the procedures in more detail. It is, of course, absolutely necessary that the tests be performed in the order given, since each test presupposes that certain substances have been eliminated.

Group A. Oils, waxes, styrene resin, vinyl acetate resin, ethyl cellulose, stearamide

A1. Evaporate solution A to dryness in the casserole.
  a. No residue = group A not present
  b. Liquid residue = oils
  c. Solid residue = waxes, styrene resin, vinyl acetate resin, ethyl cellulose, stearamide

A2. Attempt to dissolve residue A1c in acetone, warming if necessary.
  a. Residue = styrene resin
  b. Solution = waxes, vinyl acetate resin, ethyl cellulose, stearamide

A3. Evaporate solution A2b to dryness. To part of the residue, add a few cubic centimeters of 10% potassium hydroxide. Boil for several minutes. Filter. Acidify the filtrate with concentrated hydrochloric acid, heat to boiling, and allow to cool.
  a. Suspension = stearamide

A4. Attempt to dissolve the remainder of the residue from the first part of A3 in petroleum ether.
  a. Solution = wax
  b. Residue = vinyl acetate resin, ethyl cellulose

A5. To the residue A4b, add a few drops of concentrated hydrochloric acid and 1 cubic centimeter of water. Warm for 10 minutes. Place in a test tube, filtering if not clear. Add a few drops of 5% lanthanum nitrate solution and one drop of dilute iodine solution.* Make alkaline with a few drops of concentrated ammonium hydroxide solution.

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* The dilute iodine solution consists of 1 gram of iodine and 20 grams of potassium iodide in 500 cubic centimeters of water.

TABLE XVI
ORGANIC FINISHING AGENTS
GROUP A
(*Carbon tetrachloride extract*)

<table>
<tr><td colspan="6">Evaporate to dryness.</td></tr>
<tr><td rowspan="5">Liquid:
OIL</td><td colspan="5">Solid: Waxes, styrene resin, vinyl acetate resin, ethyl cellulolose, stearamide
Test with acetone.</td></tr>
<tr><td rowspan="4">Insoluble:
STYRENE
RESIN</td><td colspan="4">Soluble: waxes, vinyl acetate resin, ethyl cellulose, stearamide
Evaporate to dryness</td></tr>
<tr><td rowspan="3">Part of residue:
Dissolve in KOH, filter, acidify.
Suspension or Precipitate:
STEARAMIDE</td><td colspan="3">Part of residue:
Test with petroleum ether</td></tr>
<tr><td rowspan="2">Soluble:
WAX</td><td colspan="2">Insoluble: vinyl acetate resin, ethyl cellulose
Treat with HCl, test with $La(NO_3)_2$</td></tr>
<tr><td>Brown or blue color:
VINYL
ACETATE
RESIN</td><td>No color:
ETHYL
CELLULOSE</td></tr>
</table>

TABLE XVII
ORGANIC FINISHING AGENTS
GROUP B
(Ethyl alcohol extract)

<table>
<tr><td colspan="3">Solution:
a. Test with concentrated HCl
b. Test with KOH</td><td colspan="4">Remainder of solution: Evaporate to dryness, dissolve in boiling water, filter, cool.</td></tr>
<tr><td rowspan="2">b cloudier than a: CATIONIC FINISH</td><td colspan="2">a cloudier than b: ANIONIC FINISH
Boil HCl solution, filter, test with $BaCl_2$</td><td rowspan="2">Test with $BaCl_2$
Precipitate; SULFATED ALCOHOL</td><td rowspan="2">Test with HCl and $FeCl_3$
Green, blue, violet color: PHENOL or CRESOL</td><td colspan="2">Add $KHSO_4$, evaporate to dryness, ignite</td></tr>
<tr><td>Precipitate: SULFONATED OIL or SULFATED ALCOHOL</td><td>No precipitate: SOAP</td><td>Odor of acrolein: GLYCERIN</td><td>Odor of acetaldehyde: GLYCOL</td></tr>
</table>

TABLE XVIII
ORGANIC FINISHING AGENTS
GROUP C
(Water extract)

If solution is not clear, filter. Evaporate half of the solution to dryness.
If an appreciable residue is found, perform tests on remaining half of solution.

Test with iodine solution		Test with basic Pb $(C_2H_3O_2)_2$	Test with Fehling's Solution	Boil with HCl, test with Fehling Solution	Test with formaldehyde and $FeCl_3$	Test with a. KOH b. $HgCl_2$
Blue color: STARCH	Red or purple color: DEXTRIN	Precipitate: GUMS MOSSES ALBUMEN	Yellow, orange, red color: GLUCOSE	Yellow, orange, red color: CANE SUGAR	Violet ring: CASEIN	a. NH_3 gas b. Precipitate AMMONIUM SULFAMATE

TABLE XIX
ORGANIC FINISHING AGENTS
GROUP D
(*Dioxane extract*)

<table>
<tr><td colspan="7">Evaporate to dryness, test with toluene.</td></tr>
<tr><td rowspan="4">Soluble:
ACRYLATE RESINS</td><td colspan="6">Insoluble: phenol-formaldehyde resin, alkyd resin, cellulose nitrate, cellulose acetate, vinyl chloride resin, vinyl chloride-acetate resin
Dissolve small portion in HCl, test with carbazole.</td></tr>
<tr><td rowspan="3">Blue color:
PHENOL-FORMALDEHYDE RESIN</td><td colspan="5">No blue color: alkyd, cellulose acetate, cellulose nitrate, vinyl chloride, vinyl chloride-acetate
Test with glacial acetic acid</td></tr>
<tr><td colspan="3">Soluble: alkyd, cellulose acetate, cellulose nitrate
Evaporate to dryness</td><td colspan="2">Insoluble: vinyl chloride, vinyl chloride-acetate
Test with pyridine</td></tr>
<tr><td>Test with phenol
Red color:
ALKYD RESIN</td><td>Test with diphenylamine
Blue color:
CELLULOSE NITRATE</td><td>Test with $La(NO_3)_2$
Brown or blue color:
CELLULOSE ACETATE</td><td>Soluble:
VINYL CHLORIDE-ACETATE RESIN</td><td>Insoluble:
VINYL CHLORIDE RESIN</td></tr>
</table>

TABLE XX
ORGANIC FINISHING AGENTS
GROUP E
(Dilute hydrochloric acid extract)

Small portion Test with picric acid Precipitate: PYRIDINE DERIVATIVE	Small portion Test with carbazole		Remainder: evaporate to dryness		
			Test with formaldehyde and $FeCl_3$ Violet ring: CASEIN FORMALDEHYDE	Test with glacial acetic acid	
	Blue Color: FORMALDEHYDE RESIN	No blue color, no picric acid precipitate, but a group residue: VINYLIDINE RESIN		Soluble: UREA FORMALDEHYDE RESIN	Insoluble: MELAMINE-FORMALDEHYDE RESIN

a. Brown or blue color = vinyl acetate resin
b. No color = ethyl cellulose

Group B. Soaps, sulfonated oils, sulfated alcohols, cationic softeners, phenols or cresols, glycerin or glycols.

B1.a. To a few cubic centimeters of solution B in a test tube, add an equal volume of concentrated hydrochloric acid and then double the total volume by adding distilled water.

b. To a few cubic centimeters of solution B in a test tube, add an equal volume of 10% potassium hydroxide and then double the total volume by adding distilled water.

B1b markedly cloudier than B1a = cationic softener
B1a markedly cloudier than B1b = soap, sulfonated oil, sulfated alcohol

B2. If B1a is the cloudier, heat it to boiling. Filter, cool, and add one or two drops of 10% barium chloride solution to the filtrate.

a. No precipitate = soap
b. Precipitate = sulfonated oil, sulfated alcohol

B3. Evaporate to dryness the remainder of solution B not used in B1.

a. No residue = Group B not present

B4. Dissolve the residue from B3 in boiling distilled water, filter, and cool. To approximately one-third of the solution add 10% barium chloride solution.

a. Precipitate = sulfated alcohol

B5. To approximately one-third of the solution from the first part of B4, add one or two drops of concentrated hydrochloric acid and one drop of 10% ferric chloride solution.

a. Green, blue, or violet color = phenol or cresol

B6. To approximately one-third of the solution from the first part of B4, add several crystals of solid potassium bisulfate, evaporate to dryness, and ignite the residue.

a. Odor of acrolein = glycerin
b. Odor of acetaldehyde = glycol

Group C. Starch, dextin, sugars, gums, mosses, methyl cellulose, soluble proteins, ammonium sulfamate.

C1. If solution C is not clear, filter it. The residue is probably China clay or some other mineral weighting. Evaporate half of the solution to dryness.

a. Residue = Group C present

b. No residue = Group C not present

C2. To a few cubic centimeters of solution C, taken from the portion which was not evaporated in C1, add several drops of dilute iodine solution.

a. Blue color = starch

b. Red or purple color = dextrin

C3. To a few cubic centimeters of solution C, add several drops of basic lead acetate solution.* Allow the mixture to stand for 10 minutes.

a. Precipitate or appreciable cloudiness = gums, mosses, albumen

C4. Heat 10 cubic centimeters of Fehling's solution to boiling.† Add a few drops of solution C and boil a few minutes.

a. Yellow, orange, or red color = glucose

C5. To several cubic centimeters of solution C, add a few drops of concentrated hydrochloric acid. Heat to boiling and boil for several minutes. Add a few drops of this mixture to boiling Fehling's solution as in C4. Omit this test if starch or dextrin was found in C2.

a. Yellow, orange, or red color = cane sugar

C6. To 5 cubic centimeters of solution C in a test tube, add one drop of concentrated sulfuric acid and one drop of 3%

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* Basic lead acetate is prepared from 30 grams of lead acetate, 10 grams of litharge, and 5 cubic centimeters of water, stirred on a water bath until white. One hundred cubic centimeters of water are then stirred in. The reagent is allowed to settle and is then filtered.

† Mix 5 cubic centimeters each of Fehling's solution A and Fehling's solution B. Fehling's solution A contains 69.28 grams of crystalline copper sulfate per liter. Solution B contains 346 grams of sodium potassium tartrate and 100 grams of sodium hydroxide per liter.

formaldehyde solution. In another test tube, place 2 cubic centimeters of concentrated sulfuric acid and one drop of 10% ferric chloride solution. Pour the formaldehyde solution gently on top of the ferric chloride solution without mixing.

a. Violet ring = casein

C7. a. To several cubic centimeters of solution C, add 10% potassium hydroxide, heat gently, and immerse wet red litmus paper in the vapor.

b. To several cubic centimeters of solution C, add an equal volume of 5% mercuric chloride solution.

Blue color in a. and precipitate in b. = ammonium sulfamate

C8. If a residue is found in C1 but nothing is found in subsequent tests, the substance is probably methyl cellulose or gelatin.

Group D. Acrylate resin, phenol-formaldehyde resin, alkyl resin, vinyl chloride resin, vinyl chloride-acetate resin, cellulose nitrate, cellulose acetate.

D1. Evaporate solution D to dryness.

a. Residue = Group D present

b. No residue = Group D absent

(Vinyl chloride-acetate or cellulose acetate may be from fibers present and not from finishing materials.)

D2. Attempt to dissolve the residue from D1 in toluene, heating if necessary.

a. Solution = acrylate resin

b. Residue = phenol-formaldehyde resin, alkyd resin, vinyl chloride resin, vinyl chloride-acetate resin, cellulose nitrate, cellulose acetate

D3. Heat a small portion of residue D2b with 5 cubic centimeters of water and a drop of concentrated sulfuric acid. Cool to room temperature. Add a few drops of carbazole reagent and 1 or 2 cubic centimeters of concentrated sulfuric acid.*

* Carbazole reagent is a 0.5% solution of carbazole in 95% ethyl alcohol.

a. Blue color = phenol-formaldehyde resin
b. No blue color = alkyd resin, vinyl chloride resin, vinyl chloride-acetate resin, cellulose nitrate, cellulose acetate

D4. Attempt to dissolve the remainder of residue D2b in glacial acetic acid, warming if necessary.
a. Solution = alkyd resin, cellulose nitrate, cellulose acetate
b. Residue = vinyl chloride resin, vinyl chloride-acetate resin

D5. Evaporate solution D4a to dryness, divide residue into 3 parts.

D6. To one-third of D5, add twice as much solid phenol and one drop of concentrated sulfuric acid. Heat until brown. Cool. Dilute with water and make alkaline with 10% potassium hydroxide.
a. Red color = alkyd resin

D7. To one-third of D5, add a drop of diphenylamine reagent.*
a. Blue color = cellulose nitrate

D8. Test the remaining third of D5 as in test A5.
a. Brown or blue color = cellulose acetate.

D9. Attempt to dissolve residue D4b in pyridine or tetrachlorethane, using heat if necessary.
a. Solution = Vinyl chloride-acetate resin
b. Residue = Vinyl chloride resin

Group E. Pyridine-base waterproofing agents, vinylidene resin, casein-formaldehyde, urea-formaldehyde resin, melamine-formaldehyde resin.

E1. Retain 10 cubic centimeters of solution E; evaporate remainder to dryness.
a. Residue = Group E present
b. No residue = Group E absent

E2. To 5 cubic centimeters of solution E, add 2½ cubic centimeters of a saturated solution of picric acid.
a. Precipitate = pyridine-base waterproofing agent

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* Diphenylamine reagent is prepared by dissolving 1 gram of diphenylamine in 100 grams of concentrated sulfuric acid.

E3. To 5 cubic centimeters of solution E, add one or two drops of carbazole reagent and 1 or 2 cubic centimeters of concentrated sulfuric acid.
  a. Blue color = casein-formaldehyde resin, urea-formaldehyde resin, melamine-formaldehyde resin
  b. No blue color = vinylidene resin

E4. If E3a gave blue color, take about one-third of residue E1a; dissolve in 5 cubic centimeters of water containing one or two drops of concentrated sulfuric acid and test as in C6.
  Violet ring = casein-formaldehyde

E5. Attempt to dissolve the remainder of residue E1a in glacial acetic acid, warming if necessary.
  Solution = Urea-formaldehyde resin
  Residue = Melamine-formaldehyde resin

Quantitative Analysis

In making a quantitative determination of the amount of finishing substances, either extraction or destructive boil-off, or a combination of the two, may be used. When the finishing agent (or residual substance from processing) is a member of one of the first two groups of agents given in the preceding section, extraction with carbon tetrachloride, ethyl alcohol, or both may be used. When the finishing agent is in one of the last three groups, it is easiest to use a boil-off which destroys and dissolves the agent, but a regular extraction may be used here too. In addition to finishing agents, the determination of residual oils, greases, waxes, and soaps is also of importance and the same techniques may be used. In the manufacture of woolen and worsted goods, especially, control by extraction analysis is very important.

EXTRACTION METHODS

The extraction method of analysis depends upon separating a mixture of two substances by means of a volatile solvent in which one component of the mixture is soluble and the other is insoluble. Three general methods are available in the case of textile materials which are to be analyzed. Let us take, as examples, analyses for oil content:

Solvent Wash

Soak the sample in the solvent to remove the bulk of the oil. Place on a filter. Rinse with several volumes of the solvent. Dry, and weigh the sample. The loss in weight is oil. This method is quick and requires little apparatus but has the following disadvantages: Enormous quantities of rather expensive solvent must be used; the dissolved material is dispersed in a large bulk of solvent and cannot be easily or quickly recovered and analyzed; there is considerable fire hazard when combustible solvents are used.

Aliquot Method

Soak the sample in a known amount of solvent, take an aliquot (fractional) part of the solvent, evaporate to dryness, and weigh. Hirst uses a form of this method for determining oil in textiles: a sample of the material, weighing 10 to 20 grams, is cut up into small pieces and placed in a wide-mouthed, eight-ounce bottle with a ground-in stopper. Two hundred cubic centimeters of acetone are added and the bottle is allowed to stand with occasional shaking for 24 hours; 100 cubic centimeters of acetone are then withdrawn, filtered if necessary, and evaporated to dryness in a tared dish. The dish is cooled in a desiccator, and then weighed. The weight of the extract multiplied by 2 gives the total quantity of oil. A number of tested oils gave 99% recovery in this manner, including oxidized oils, and the amount of soap dissolved was negligible.

Use of Special Extraction Apparatus

Either the Soxhlet apparatus or the rubber extraction apparatus may be used. This is the usual method and will be discussed here in detail.

The Soxhlet apparatus (Fig. 49) consists of three parts connected by ground-glass joints: the flask, the extraction tube, and the condenser. The material to be tested is placed in the extraction tube, and the solvent to be used is placed in the flask; the flask is heated, preferably with an electric hot plate. On heating, the vaporized solvent passes up through the vapor tube (shown on the left side of the extraction tube in Fig. 49) and is condensed to a liquid in the condenser, from where it drips down on the sample in the extraction tube. When enough solvent has condensed and fallen on the sample to fill the extraction tube up to the top of the siphon (shown on the right side of the extraction tube in Fig. 49), it siphons over into the flask and the cycle starts over again. While the solvent is in contact with the sample, it dissolves much of the impurity which passes down into the flask. Another lot of pure solvent then condenses and removes more impurity. The Soxhlet apparatus, then, finally gives us the sample free of impurity, and the dissolved material is finally all concentrated in the relatively small amount of solvent in the flask, from which it may be recovered by evaporation for weighing and analyzing, if required.

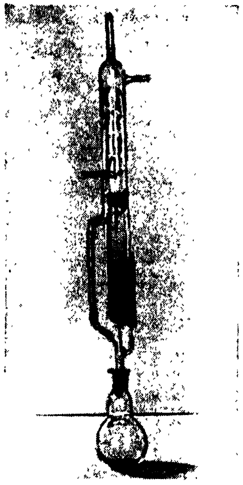

FIG. 49. Soxhlet Extraction Apparatus

The rubber extraction apparatus is less frequently used. It works on the same principle, but the extraction tube is suspended in the flask from a coil of metal tubing which acts as a condenser. The rubber apparatus is less expensive than the Soxhlet, but it is not so safe for use with combustible solvents. The Soxhlet apparatus may be considered as the standard apparatus for textile purposes; the rubber extraction apparatus is specified by the A.S.T.M. for determining oil and grease in knit goods with carbon tetrachloride, but all other determinations specify the Soxhlet.

If the sample to be tested is in a finely divided state or if it contains a large amount of mechanical dirt or short, loose fibers (as in some shoddies), an extraction thimble should be used to hold the sample. This may be a tube made of porous paper, an alundum tube, or a glass tube with a coarse fritted-glass bottom. The sides of the thimble, obviously, must be higher than the siphon tube, otherwise the small particles might float out into the extraction tube and ultimately into the flask.

The actual routine of carrying out an extraction varies according to equipment available, e.g., whether the oven and analytical balance are separate units or are combined (see Chapter 2); it also varies as to whether the chemist considers weighing the extract or weighing the loss of the sample as the most accurate method.

If a separate oven and analytical balance are used, it is necessary to use a weighing bottle which adds another weighing to the process with another source of error. If the extract is weighed, time is required to evaporate off the solvent and, of course, the flask must have been previously dried and weighed; some time may be saved by not weighing the sample before extraction but drying and weighing the extracted sample afterward (at the same time that the extract is being dried and weighed) and adding the weight of the extract to the weight of extracted sample to obtain the sample weight. The following cases, therefore, may exist and the formula for each case is given:

Let

A = weight of weighing bottle

B = weight of weighing bottle + sample

C = weight of weighing bottle + extracted sample
S = weight of sample
E = weight of extracted sample
F = weight of flask alone
G = weight of flask + extract

Using separate oven and analytical balance

1. Weighing extract
    a. Weighing sample before extraction
    Per cent loss on extraction $= \frac{C - F}{B - A} \times 100$
    (4 weighings)
    b. Weighing extracted sample and adding extract
    Per cent loss on extraction $= \frac{G - F}{(C - A) + (G - F)} \times 100$
    (4 weighings)
2. Weighing loss of weight of sample
    c. Per cent loss on extraction $= \frac{B - C}{B - A} \times 100$
    (3 weighings)

Using combined oven and analytical balance

1. Weighing extract
    d. Weighing sample before extraction
    Per cent loss on extraction $= \frac{G - F}{S} \times 100$
    (3 weighings)
    e. Weighing extracted sample and adding extract
    Per cent loss on extraction $= \frac{G - F}{E + (G - F)} \times 100$
    (3 weighings)
2. Weighing loss of weight of sample
    f. Per cent loss on extraction $= \frac{S - E}{S} \times 100$
    2 (weighings)

It will be noted that a combined oven and balance requires fewer weighings which makes for greater accuracy and speed. Whether the weight of extract in the flask or the loss in weight of the sample should be determined is largely a matter of prejudice. If the extract is to be analyzed or qualitative tests are to be carried out on it, of course, the extract should be separated out and weighed, but if only the amount of extracted matter is to be determined, either method may be used. It is sometimes stated that weighing the extracted matter is more direct; but this is a fallacy, since in one case we are measuring an increase in weight of a flask and in the other case we are measuring a decrease in weight of a sample. One difference might show up in the case of an easily oxidized oil. If we heat the sample before extraction, we might oxidize the oil and make it less soluble in the solvent so that the loss in weight of the sample by method (c) or (f) would be too small; this would not apply to weighing the extract if we use method (b) or (e). However, the same oil must be heated in the flask in method (b) or (e) to drive off the last traces of solvent. This may now oxidize and increase the weight of extract. In the case of an oxidizable oil, therefore, one method tends to be too low and the other method tends to be too high. The usual method is to weigh the extract, but there is no reason for it except custom, and it has been found that better checks and more consistent results have been obtained by measuring the loss in weight of the sample. Whichever method is used, as large a sample as the apparatus will hold without too tight packing should be used, because in either method we are measuring a small amount of extract by the change in weight of a relatively heavy sample or flask.

The solvent to be used in an extraction depends upon what we are trying to determine. Oils, fats, and waxes are usually determined with ethyl ether, petroleum ether, or carbon tetrachloride; ethyl ether may be considered the standard for this work but carbon tetrachloride is used more generally than ether in plant work because of less fire hazard and less loss by evaporation. Carbon tetrachloride gives a larger amount of extract, in the case of wool, than ether because of some solvent action on soaps which may be present. Petroleum ether, on the other hand, gives a lower value than ethyl ether because oxidized fatty acids are much more soluble in ethyl ether than in petroleum ether. In the case of raw cotton, the wax may be determined with one of the above solvents, but if the combined cotton wax and resin are to be determined then chloroform, benzene, or trichlorethylene should be used. In the case of other members of Group A (preceding section), of course, carbon tetrachloride must be used.

Soaps of sodium or potassium are determined, after the oil is removed, by an extraction with ethyl alcohol. Absolute ethyl alcohol has been suggested by some workers, but 95% ethyl alcohol gives sufficiently accurate results and may be considered the standard. If the extract is determined by weighing the loss in weight of the textile sample and an alcohol extraction is made, the sample, after being extracted, should be immersed in water a few minutes, then squeezed, dried, and weighed.

If this water treatment is not given, some of the alcohol will be retained by the sample even in the drying oven and will increase the apparent weight of the extracted sample. The alcohol extract may also contain oxidized oils which are not very soluble in ether, glycerin, and traces of zinc chloride or magnesium chloride if present in large amounts in the sample. It may also contain other members of Group B of the preceding section.

The insoluble soaps, such as calcium or magnesium soaps, are not appreciably removed by alcohol and may be determined by soaking the sample (after the ether and alcohol extractions) for 1 hour in dilute hydrochloric acid, washing, drying, and extracting again with ether. The loss in weight of the sample then represents soaps of the heavy metals and alkaline earth metals. Gilmore has suggested a complete removal of all these constituents—oils, fats, waxes, resins, soaps, and calcium or magnesium soaps—by extraction with a benzene-alcohol mixture (9 parts benzene, 1 part alcohol) followed by a regular chemical analysis of the extract.

The time required for an extraction has been stated by various workers as anywhere from 1 hour to 24 hours. A better method is to extract for a given number of siphonings. For ether or alcohol, the author has always found twelve siphonings to be sufficient; twenty siphonings are sometimes recommended but there is an insignificant difference in the results, and in the case of alcohol, at least, there is evidence to indicate that too great a number of siphonings is more in error than too few.

Table XXI gives the range of extractable matter in various types of cotton. Table XXI also gives the A.S.T.M. maximum permissible extracted matter for certain textile materials.

TABLE XXI
EXTRACTED MATERIAL IN COTTON AND WOOL

Material	*Solvent*	*Per Cent Extracted*
Cotton, American	Carbon tetrachloride	0.35–0.51
Cotton, Egyptian	Carbon tetrachloride	0.35–0.43
Cotton, Sea Island	Carbon tetrachloride	0.5 (approx.)
Cotton, South American	Carbon tetrachloride	0.29–0.54
Cotton, Indian	Carbon tetrachloride	0.30–0.42
Cotton, boiled-out	Carbon tetrachloride	0.1–0.2
Cotton goods to be rubberized (A.S.T.M.)	Carbon tetrachloride	1.0 (max.)
Wool, scoured, 64's and finer (A.S.T.M.)	Ether	1.0 (max.)
Wool, scoured, coarser than 64's (A.S.T.M.)	Ether	1.5 (max.)
Wool, scoured (A.S.T.M.)	Alcohol (after ether)	1.0 (max.)
Woolen yarn, scoured, spun in oil (A.S.T.M.)	Ether and alcohol	7.0 (max.)
Woolen yarn, semi-scoured, spun in oil (A.S.T.M.)	Ether and alcohol	15.0 (max.)
Woolen yarn, dry spun (A.S.T.M.)	Ether and alcohol	2.0 (max.)
Worsted yarn, oil spun (A.S.T.M.)	Ether and alcohol	4.0 (max.)
Worsted yarn, dry spun (A.S.T.M.)	Ether and alcohol	1.5 (max.)

The natural cotton wax is composed of a large number of substances of which gossypyl alcohols are the most important. The cotton wax has a melting point of 75–80° C., an iodine value of 22–32, and contains 54–64% of unsaponifiable matter. These constants may be used to distinguish this wax from the substances which may be applied to cotton in sizing and finishing; the most common of these are tallow, spermaceti, castor oil, mineral oil, paraffin wax, Japan wax, and sometimes other waxes or some of the more recent fatty alcohol products or sulfonated oils.

In discussing the analysis of the ether extract, we may consider the ether extract of wool as the most common as well as the most complex example. The ether extract should theoretically contain:

1. All the free fatty acids
2. All the oils, fats, and waxes, including any residual wool grease
3. No soaps

The ether extract actually obtained will differ from the above in the following manner:

1. All the free fatty acids will be present if the wool is a normal soap-and-soda scoured specimen and ethyl ether is used. If petroleum ether is used, the oxidized fatty acids will not all be present. In addition, free acids of equivalent weight 327 may be present from the wool grease.
2. Oxidized oils will not all be present, nor will all of the residual wool grease.
3. A small amount of soap may be present, probably in the form of an *acid soap*.

Tests which may be carried out on the ether extract include the following:

1. Test for rancidity (Kreis test)

One drop of the oil is placed on a porcelain plate. One drop of concentrated hydrochloric acid (specific gravity 1.19) is added and the drops are mixed with a fine glass rod. One drop of a 0.1% solution of phloroglucinol in ether is added. A red color indicates rancidity.

2. Test for mineral oil

This is best tested for by the refractometer or by ultraviolet radiation.

3. Test for rosin or rosin oil (Liebermann-Storch test)

A small amount of sample is warmed with acetic anhydride in a small test tube until a clear solution is obtained. A drop or two of this solution is then placed on a porcelain plate and allowed to cool. A drop of sulfuric acid (specific gravity 1.53) is then placed on the plate near the other drop and the two drops are allowed to coalesce. A violet color indicates rosin or rosin oil.

4. Test for cottonseed oil (Halphen test)

Two cubic centimeters of oil in a small test tube are dissolved with 2 cubic centimeters of amyl alcohol and 2 cubic centimeters of 1% solution of sulfur in carbon bisulfide. The test tube is immersed in a boiling water bath for about 10 minutes. An orange or crimson color indicates cottonseed oil.

5. Test for sesame oil (Baudouin test)

Two cubic centimeters of oil are placed in a small test tube, one drop of a 1% alcoholic solution of furfural and 2 cubic centimeters of concentrated hydrochloric acid are added and shaken well. A rose to crimson color in the aqueous liquid indicates sesame oil.

6. Test for peanut oil (Bellier test)

Two solutions are necessary: alcoholic potash, 8.5 grams of potassium hydroxide in 70% ethyl alcohol made up to 100 cubic centimeters; acetic acid, 120 cubic centimeters of 36% acetic acid diluted to 150 cubic centimeters (1.5 cubic centimeters of this acid should exactly neutralize 5 cubic centimeters of alcoholic potash). To 1 gram of oil in a test tube 5 cubic centimeters of alcoholic potash solution are added. This mixture is boiled gently over a small flame until saponification is complete (about 2 minutes). Evaporation should be avoided as much as possible. One cubic centimeter of acetic acid is added, mixed well, and the whole is cooled 30 minutes in tap water, with occasional shaking. Fifty cubic centimeters of 70% alcohol containing 1% by volume of concentrated hydrochloric acid are added, shaken well, and the test tube is placed in tap water for 1 hour. A clear opalescent liquid is obtained if peanut oil is not present, a precipitate is obtained if peanut oil is present.

7. Test for teaseed oil

Seven drops of oil are added to a previously cooled mixture of 0.8 cubic centimeter of acetic anhydride, 15 cubic centimeters of chloroform, and 0.2 cubic centimeter of concentrated sulfuric acid. After standing 2 or 3 minutes, 10 cubic centimeters of anhydrous ether are added and mixed well. A pink coloration indicates teaseed oil.

8. Test for unsaponifiable matter and animal and vegetable oils

The oil is saponified with alcoholic potash and is then shaken with ether to dissolve the unsaponifiable matter which may be recovered by evaporating the ether extract. This unsaponifiable matter will contain cholesterol if animal oils are present or phytosterol if vegetable oils are present, these may be identified by the melting points of their acetates. The unsaponifiable matter is boiled with acetic anhydride (an oily layer on the surface at this point indicates hydrocarbons). The excess acetic anhydride is evaporated off on the water bath. The residue is dissolved in hot alcohol, crystallized, washed with 95% alcohol, recrystallized from alcohol, and the melting point determined. The melting point of cholesterol acetate is 114–115° C. and that of phytosterol acetate is 125–137° C.

9. Test for oxidized fatty acids

Any ether extract which is not soluble in petroleum ether may be considered as oxidized fatty acids.

The alcohol extract obtained after an ether extraction should theoretically contain:

1. No fatty acids
2. No oils, fats, or residual wool grease (except potassium soaps from the suint)
3. All of the soaps

Actually, the normal soap-and-soda scoured wool will contain:

1. An appreciable amount of a water-soluble acid having an equivalent weight of 254.
2. A fairly large amount of residual wool grease, which is ether soluble after removal from the wool but which is not removed from the wool by ether extraction, also any oxidized oils.
3. Practically all of the soap.
4. Traces of salts such as potassium chloride and potassium sulfate from the wool, also some zinc chloride or magnesium chloride if these have been present in the sample in fairly large amounts.
5. Glycerin, if present on the original sample.

DESTRUCTIVE BOIL-OFF METHODS

To remove finishing materials of Groups C, D, and E, extraction with the solvents given for group reagents in the identification may be used, but it is easier and quicker to use one of the methods of destruction and boil-off. Three methods have been given in the literature:

1. The alkali-acid or scouring method
2. The acid-alkali method
3. The enzyme or malting method

The scouring method is now never used. The enzyme method can be used only on the starches and gums, where it is considered most accurate, although the author has not so found it. The acid-alkali method is applicable to all finishes in Groups C, D, and E and is at least as good as the enzyme method for starch. Where large amounts of synthetic resin are present, a second acid-alkali treatment may be necessary to complete the removal of the finish.

If a cotton sample has been through any sort of a scouring process (boil-out, bleaching, dyeing, or mercerizing) there is no correction factor to be applied, but if raw cotton has been used it is necessary to apply a correction factor which is subtracted from the loss on desizing to give finishing materials. These correction factors, however, are so variable in amount that a good average is not possible. It is, therefore, safer to report the *loss on de-sizing* and state the method used, unless a sample of the cloth before finishing or sizing is available as a blank.

Scouring Method

The scouring method is carried out as follows: A five-gram sample is dried and weighed and is then treated for 1 hour at 80° C. with 200 cubic centimeters of 1% sodium hydroxide, after which it is well rinsed and wrung out in hot water. It is then heated for 1 hour at 80° C. in 200 cubic centimeters of $\frac{1}{2}$% hydrochloric acid, after which it is rinsed, boiled for $\frac{1}{2}$ hour in distilled water, dried, and weighed.

Acid-alkali Method

The acid-alkali method is given with many small variations. The A.S.T.M. method is carried out as follows: A sample of fabric of 1 square foot is dried and weighed. It is then boiled 30 minutes in $\frac{1}{2}$% hydrochloric acid (the acid should be boiling when the sample is put in), removed and rinsed thoroughly. It is then boiled 30 minutes in 1% sodium carbonate, removed, rinsed, dried, and weighed.

Enzyme Method

The enzyme method is also used in many slightly different forms, the A.S.T.M. and A.A.T.C.C. methods being practically identical. The A.A.T.C.C. method is carried out as follows: A ten-gram sample is dried and weighed, rinsed in hot distilled water and is then immersed in twenty times its weight of a 3% solution of a starch-solubilizing enzyme (such as Diastafor) at 60° C. It is squeezed three times with the solution to insure proper wetting-out. It is digested for 1 hour in this solution at 60° C. (temperature must not be allowed to go above 60° C. or the enzyme action will be destroyed), after which it is rinsed well in hot water, all loose fibers being collected on a 100-mesh sieve or a Gooch crucible. The sample is then boiled for 1 hour in distilled water and is rinsed again, after which it is squeezed, dried, and weighed.

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Chapter 14

FIBER MIXTURES

Qualitative Analysis

The identification of fibers by purely chemical means is so complex as to be almost an impossibility, although in a great many cases a strong degree of probability may be established. The principal difficulties to which this uncertainty is due are:

1. Many times a test indicates a group of fibers rather than a single fiber. For example, a chemical test might show that a certain fiber was an animal hair but there would be no chemical method of determining which animal's hair.

2. Finishing materials may affect the tests and give false results. For example, casein will give the same test as animal fibers and starch may give the same test as swollen cellulose fibers.

3. Dyes interfere with most of the color reactions and even the trace of dye left after stripping may do the same.

4. If a solubility test is used, a small amount of one constituent of a mixture may be dissolved with no noticeable effect. For example, if a mixture were 5% silk and 95% wool, the silk might be dissolved in concentrated hydrochloric acid with no noticeable change in the mixture.

5. Degraded or modified fibers may give the reactions of another fiber. For example, a wool shoddy containing a small amount of carbonized cotton might completely dissolve in 5% potassium hydroxide.

6. New fibers are continually being introduced which have some properties of older fibers. For example, soybean fiber gives the reactions of casein fiber and would normally not be differentiated from it.

7. Some tests depend upon the detection of an impurity in the fiber, and if the fiber is made very much purer it may no longer give the test. For example, hemp ordinarily gives a test for lignin and so falls into the group with jute, but if sufficiently purified in processing, it no longer gives the test and falls in the group with cotton and flax.

In view of the above-mentioned facts, the only safe way to analyze a mixture of fibers is by using the microscope in conjunction with the tests to be described later in this chapter and to confirm every reaction by other reactions or by the microscope.

The tests usually carried out may be classified as follows:

1. Burning tests
2. Color tests
3. Solubility tests

These will be discussed in more detail.

4. Dyeing or staining tests. These are too uncertain because they are too likely to be affected by traces of impurities and slight variations in the method of procedure.

5. Microscopic examination. This will be omitted in this chapter and will be taken up in another section.

Since we are to attempt to identify the fibers by chemical means, we should classify the fibers according to chemical composition.

Classification of Fibers

A. Mineral or part-mineral fibers
    1. Glass
    2. Asbestos
    3. Weighted silk
B. Artificial plastic fibers
    1. Acetate rayon (except the saponified type)
    2. Vinyl fiber
    3. Nylon

C. Natural and artificial protein fibers
  1. Natural
    a. Silks (true silk, tussah)
    b. Hairs (wool, mohair, camel hair, etc.)
  2. Artificial
    a. Casein fiber
    b. Soybean fiber
    c. Peanut, zein, etc. fibers
D. Natural and artificial cellulose fibers
  1. Artificial (swollen cellulose)
    a. Mercerized cotton
    b. Cupra rayon
    c. Viscose rayon
    d. Saponified acetate rayon
  2. Natural
    a. Lignocellulose (jute, hemp, kapok, etc.)
    b. Cellulose (cotton, flax, ramie, etc.)

CHEMICAL TESTING

Table XXII gives a summary of the chemical tests which may be used for identification. For the sake of compactness, the table contains only the fibers commercially available and commonly used for textile fabrics. The details of the tests follow.

The sample to be tested should be freed of all finishing agents by treatment with boiling dilute hydrochloric acid such as was used in determining finishing agents in the previous chapter. Unless it is known that a fabric or yarn is composed of one fiber only, the fabric should be dissected and the yarns untwisted to obtain fibers of each kind present.

The first test is the burning test. Fibers are brought up to a match flame slowly and it is observed whether they burn or melt. If the fibers do not burn or melt, they are glass or asbestos. If they burn but the ash retains the shape of the fiber, they are weighted silk. If they burn or melt they are organic fibers and the odor may give an indication of which fiber they are. However, it is best not to use odor as a positive test. If glass or asbestos is

TABLE XXII
QUALITATIVE ANALYSIS OF FIBERS

<table>
<tr><td colspan="13">*All Fibers—Hold in Match Flame*</td></tr>
<tr><td colspan="2">Do not burn:
ASBESTOS
GLASS
Hold in Bunsen flame</td><td>Burn, ash retains shape:
WEIGHTED SILK</td><td colspan="10">Fibers burn, with or without odor, or melt to ball
Test with acetone at room temperature</td></tr>
<tr><td>Melt:
GLASS</td><td>Do not melt:
ASBESTOS</td><td></td><td colspan="2" rowspan="2">Soluble:
ACETATE, VINYL
Test with cold glacial acetic acid</td><td colspan="8">Insoluble
Test with boiling glacial acetic acid</td></tr>
<tr><td></td><td></td><td></td><td>Soluble:
NYLON</td><td colspan="7">Insoluble:
Boil in 5% KOH</td></tr>
<tr><td></td><td></td><td></td><td>Soluble:
ACETATE</td><td>Insoluble:
VINYL</td><td></td><td colspan="3">Soluble: PROTEINS
Test with cold, concentrated HCl</td><td colspan="4">Insoluble: CELLULOSES
Strip dye, test with $ZnCl_2$-I_2</td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td>Soluble:
SILKS</td><td colspan="2">Insoluble:
HAIRS, AZLONS
Test with 10% KOH at 50° C, 30 minutes</td><td rowspan="3">Blue
MERCERIZED COTTON
CUPRA
VISCOSE</td><td colspan="3">No color:
COTTON, FLAX, JUTE
If undyed, test with aniline sulfate</td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>Soluble:
HAIRS</td><td>Insoluble:
CASEIN
SOYBEAN</td><td>Yellow:
JUTE</td><td colspan="2">No color:
COTTON, FLAX
Moisten fiber with fingers</td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>Tip moves clockwise:
FLAX</td><td>Tip moves counterclockwise:
COTTON</td></tr>
</table>

found, other samples of the same fibers are similarly brought up to the side of a Bunsen flame; if the fibers now melt, they are glass; if they do not melt, they are asbestos.

If it has been determined that the fibers are organic, a fresh sample is placed on a watch glass and a few drops of acetone at room temperature are added. If the sample now dissolves, it is acetate rayon or vinyl fiber. A fresh sample of the same fibers is similarly tested with glacial acetic acid at room temperature; acetate rayon dissolves, but vinyl fiber does not.

If these two fibers have been eliminated, some of the original sample is tested on a watch glass or in a test tube with boiling glacial acetic acid. Nylon is soluble; other fibers are not. Both acetate and vinyl fibers would dissolve here also, but they have already been determined.

The next test distinguishes between the protein fibers and the cellulosic fibers. A sample of the fibers is boiled in a watch glass or test tube with 5% potassium hydroxide solution. If the fiber dissolves, it is a natural or artificial protein fiber. If the fiber is insoluble, it is a natural or artificial cellulosic fiber.

If the fiber is found to be protein, a fresh sample is treated on a watch glass with concentrated hydrochloric acid at room temperature. If the fiber dissolves, it is a silk. (True silk dissolves rapidly, tussah silk slowly.) If the fiber does not dissolve, it is a hair or an artificial protein. In the latter case, a sample of the fiber is treated in a test tube with a 10% potassium hydroxide solution at 50° C. for 30 minutes. This is best done in a water bath. If the fiber now dissolves, it is a hair such as wool, mohair, camel hair, etc. If it does not dissolve, it is an artificial protein fiber such as casein fiber, soybean fiber, peanut fiber, etc.

If the sample was found to be cellulosic by the 5% potassium hydroxide test, the dye is stripped from it by a mild hydrosulfite solution and it is then tested for swollen cellulose by means of zinc chloride-iodine reagent. The solution and test is as follows: Solution A is 280 grams of zinc chloride in 300 cubic centimeters of water. Solution B is 1 gram of iodine and 20 grams of potassium iodide in 100 cubic centimeters of water. The reagent is prepared by adding six drops (0.2 cubic centimeter) of Solution B to 20 cubic centimeters of Solution A. The previously wet-out and squeezed sample is immersed in the mixture at room temperature for 3 minutes and is then viewed while still in the solution. Swollen celluloses, such as mercerized cotton, cupra rayon, viscose rayon, and saponified acetate rayon, are colored blue; other cellulose fibers are not colored. It is always well to run a sample of bleached but unswollen cotton and a sample of mercerized cotton along with the unknown in this test to insure that the reagent is of the correct strength and properly mixed.

If the sample, now known to be cellulosic, is not dyed, another test may be performed. The sample, on a watch glass, is moistened with a 2% aniline sulfate solution. If the sample is colored yellow, it is a lignocellulose such as jute or hemp. If the sample is not colored, it is a cellulose such as cotton, flax, or ramie. In these last two cases, a fairly reliable test is to hold a specimen fiber at one end and with moist fingers, wet the free end. In the case of flax and ramie, the free end will revolve clockwise. In the case of cotton and hemp, the free end will revolve in a counter-clockwise direction.

Reference to the table will show that we have now identified definitely certain fibers, others however still remain in groups of chemically similar fibers, these are:

1. Silks (true silk, tussah)
2. Hairs (wool, mohair, etc.)
3. Swollen cellulose (mercerized cotton, viscose rayon, cupra rayon, saponified acetate rayon)
4. Lignocellulose (kapok, jute, hemp)
5. Cellulose (cotton, flax, ramie)

At this time, the microscope should be used, but if one is not available the following tests may be employed.

The silks, if undyed, may be differentiated by color; tussah silk is a brown color, whereas degummed true silk is white. If the fiber is dyed or undyed, a fair idea may be obtained from its behavior in cold concentrated hydrochloric acid; silk dissolves rapidly, tussah much more slowly. In the same way, boiling 1% sodium hydroxide dissolves true silk very rapidly, whereas tussah silk requires at least 30 minutes to dissolve.

The hairs cannot be differentiated by any chemical tests. If undyed, camel hair can be distinguished from wool by its brown color and an expert can often distinguish one hair from another by the appearance.

Of the swollen celluloses, three are rayons and the other is mercerized cotton. If the specimen is continuous filament, of course, it is one of the rayons. The lustrous appearance of the rayons, also, is usually enough to distinguish it from the mercerized cotton. The differentiation of cupra and viscose rayons by any other means than the microscope is uncertain but the following two methods may be tried. Wilson's method consists of treating about 0.2 gram of sample with 10 cubic centimeters of concentrated sulfuric acid in a test tube; both cupra and viscose dissolve, cupra with a yellow or brownish color immediately and a brownish-yellow after 40 minutes, viscose with a reddish-brown color which is a rusty brown after 40 minutes. The sulfide test of the A.S.T.M. may also be used: 5 grams of sample are placed in a conical flash with a capacity of 250 cubic centimeters together with 100 cubic centimeters of water and 3 cubic centimeters of concentrated sulfuric acid. The mouth of the flask is covered with a piece of lead acetate paper, and the flask is allowed to stand at 90 to 95° C. for 4 hours. Viscose stains the paper yellowish to brown or possibly black, cupra does not stain the paper.

Of the lignocelluloses, kapok is never found in the form of yarn or cloth but only in bulk, it may be recognized by the silky feel, the resistance to wetting by water, and by the relatively short staple. By immersion in chlorine water for 1 minute, spreading on a white porcelain dish, and the addition of several drops of ammonia solution, jute and kapok are given a red or violet color while hemp is given at most a brown color.

The cellulose fibers (cotton, flax, and ramie) cannot be chemically differentiated. The staple of the cotton is usually much shorter than either flax or ramie. Ramie is usually somewhat stiffer than either cotton or linen. For complete verification of the identity, the microscope should be used.

The continual introduction of new synthetic fibers should be borne in mind, and the above tests must be modified whenever necessary. It must be emphasized again that for a definite and certain knowledge of what fibers are present, especially in a rather complex mixture, the microscope must be used.

Quantitative Analysis

The quantitative methods for analyzing mixtures of fibers may be grouped as follows:

MECHANICAL ANALYSIS

This consists in picking out all of one fiber from the mixture of two or more fibers and weighing all of each fiber separately. An example would be a case in which the warp threads were cotton and the filling threads wool; in this case each set of yarns may be weighed and its percentage calculated. This method is not applicable to blends of fibers, however.

MICROSCOPIC ANALYSIS

This consists in counting the number of each kind of fiber in a mixture. To be accurate, the number of fibers should be multiplied by their average weight to get the percentage by weight. This method, therefore, gives only approximate results and is used only in cases where no other method is available. An example of a case where this method would be used is a wool-mohair blend.

PROXIMATE ANALYSIS

This is the determination of some chemical or physical constant of the mixture and the calculation, from this, of the composition of the mixture. Examples of this type of analysis which have been used are:

Determination of specific gravity of a mixture and calculation, from it, of the composition. This has been suggested for cotton-wool mixtures.

Determination of the fluidity of a mixture of cotton and viscose and calculating the percentage composition on the basis that the fluidity of cotton in $\frac{1}{2}\%$ solution is 1 to 2 and of viscose is 36 to 40.

Determination of the nitrogen content (by the Kjeldahl method) of a mixture of animal and vegetable fiber and calculation of the composition. This is rather inaccurate because of the variable nitrogen content of the animal fibers, also because of the presence of nitrogen-containing dyes, finishing compounds, or weighting.

Determination of the P_2O_5-content of a mixture of wool and casein fiber and calculation of its composition (casein fiber = 1.80% P_2O_5). This is more tedious and no more accurate than other methods.

Proximate analyses tend to be inaccurate, in general, because of the variation in the constant determined in one or more of the constituents of the mixture.

CHEMICAL ANALYSIS

This consists in weighing a sample, dissolving out one component, weighing the residue, and continuing the process until the composition is known. Nearly always a reagent which removes one fiber has some effect on another, so that in general correction factors are necessary. This method of analysis is in most common use and will be considered in more detail.

The reagents used for chemical methods of analysis for the common fiber mixtures are as follows:

1. Acetate rayon and any other fiber mixtures
    *a. Acetone
    *b. Glacial acetic acid

2. Cotton-wool or rayon-wool mixtures
   - *a. Potassium hydroxide
   - b. Aluminum chloride
   - *c. Sulfuric acid
3. Silk-cotton or silk-wool mixtures
   - *a. Hydrochloric acid
   - b. Ammoniacal nickel oxide
   - *c. Concentrated calcium thiocyanate
   - d. Basic zinc chloride
4. Silk-rayon mixtures
   - a. Dilute calcium thiocyanate
   - b. Copper glycerol hydroxide
   - c. Ammoniacal nickel oxide
   - *d. Potassium hydroxide
5. Cotton-rayon mixtures
   - *a. Concentrated calcium thiocyanate
   - b. Semi-saturated chromic acid
   - c. Sodium zincate

Many of the above reagents give very erratic results. The reagents marked by an asterisk are the most satisfactory and are sufficient to take care of any mixture of the common fibers. These methods will be given in detail.

Acetone Method

Dry and weigh the sample (about 5 grams). Warm in a beaker with 250 cubic centimeters of acetone for 30 minutes. Filter through a Gooch filter or 100-mesh screen. Wash twice with acetone, then with water, dry, and weigh. Acetate rayons, with a few exceptions, are completely soluble; other fibers are insoluble. If the acetate rayon is found by a previous test to be not completely soluble in acetone, use the glacial acetic acid method.

Glacial Acetic Acid Method

Dry and weigh the sample (about 5 grams). Drop into 250 cubic centimeters of boiling glacial acetic acid and boil for

20 minutes. Filter through a Gooch filter or 100-mesh screen. Wash with glacial acetic acid. Wash with water. Wash with dilute ammonia. Wash with water. Dry and weigh. This treatment dissolves all acetate rayons and nylon but affects other fibers somewhat. (See Table XXI.)

Five Per Cent Potassium Hydroxide Method

Dry and weigh the sample (about 5 grams). Drop into 250 cubic centimeters of boiling 5% potassium hydroxide and boil for 15 minutes. Filter through a Gooch filter or 100-mesh screen. Wash with potassium hydroxide solution. Wash with water. Wash with dilute acetic acid. Wash with water. Dry and weigh. This dissolves wool and silk. The residue is cotton, rayon, or nylon. Acetate rayon is partly soluble to such a variable extent that no correction factor can be given; it should, however, have been already removed by acetone or glacial acetic acid. In routine work, where the expense of the reagent is a consideration, sodium hydroxide may be used instead of potassium hydroxide. For important work or referee work, the potassium hydroxide should be used.

Ten Per Cent Potassium Hydroxide Method

Dry and weigh the sample (about 5 grams). Drop into 250 cubic centimeters of 10% potassium hydroxide solution which has been brought to 50° C. in a water bath. Maintain the sample and solution at 50° C. for 30 minutes, stirring every 5 minutes. Filter through a Gooch filter or 100-mesh screen. Wash with potassium hydroxide. Wash with water. Wash with dilute acetic acid. Wash with water. Dry and weigh. This procedure is intended to separate wool and casein fiber and should be used only when both of these fibers are present in a mixture.

Hydrochloric Acid Method

Dry and weigh the sample (about 5 grams). Drop into 250 cubic centimeters of concentrated hydrochloric acid at room temperature for 15 minutes. Filter through a Gooch filter or

TABLE XXIII
QUANTITATIVE ANALYSIS OF FIBERS
Solubility in Various Reagents

	Acetone Method	*Glacial Acetic Acid Method*	*Five Per Cent Potassium Hydroxide Method*	*Ten Per Cent Potassium Hydroxide Method*	*Cold, Concentrated Hydrochloric Acid Method*	*Calcium Thiocyanate Method 1.36 Sp. Gr.*	*A.A.T.C.C. Sulfuric Acid Method*
WOOL	0%	1.5%	100%	100%	0.5%	2%	0% (Reworked wool = 4%)
(Mohair)							
(Camel hair)							
CASEIN FIBER	0%		100%	12.5%			
(Soybean fiber)							
SILK	0%	5%	100%	Partly soluble	100%	100%	100%
(Tussah)							
ACETATE RAYON	100%*	100%	Partly soluble	Partly soluble	Partly soluble	100%	100%
NYLON	0%	100%	0%	0%	100%	0%	100%†
VISCOSE RAYON	0%	3%	5%	2%	Partly soluble	100%	100%
(Cupra)							
(Saponified acetate)							
COTTON	0%	2%	3%	0%	4%	2.5% (Mercerized cotton = 4%)	100%
(Linen)							
(Ramie)							

*A few acetate rayons are not completely soluble in acetone. If the sample (by test) is one of these, the glacial acetic acid method should be used; otherwise the acetone method is preferable.

† In wool-nylon mixtures, on washing with water, nylon may precipitate on the wool; to prevent this, wash first with 70% sulfuric acid and then with water.

100-mesh screen. Wash with concentrated hydrochloric acid. Wash with water. Wash with dilute ammonia. Wash with water. Dry and weigh. This treatment dissolves silk and nylon but not cotton or wool. Rayons and acetate rayons are partially dissolved and no correction factor can be given.

Calcium Thiocyanate Method

The official method of the A.S.T.M. and the Bureau of Standards is too critical and requires special apparatus. The following method is simpler and as accurate. Dry and weigh the sample (about 2 grams). Prepare a solution of calcium thiocyanate with a specific gravity of 1.36 and place 200 cubic centimeters of the solution, in a flask, in a boiling water bath or steam bath so that the temperature is 100° C. Do not boil the solution. Drop the sample into the solution and leave for 1 hour, stirring it well every 15 minutes. Filter through a Gooch filter. Wash with calcium thiocyanate solution. Then wash well with water. Dry and weigh. The calcium thiocyanate may be recovered by diluting with water until the dissolved fiber is precipitated, and concentrating again to a specific gravity of 1.36. This treatment dissolves silk, rayon, and acetate rayon but not wool, cotton, or nylon.

Sulfuric Acid Method

Older methods were very unsatisfactory, but the method of the A.A.T.C.C. is very satisfactory. Dry and weigh the sample (about 2 grams). Immerse the sample in 200 cubic centimeters of a boiling 1% solution of sulfuric acid for 7 to 10 minutes. Transfer to a Gooch filter and remove the excess acid by suction. Place the sample in 200 cubic centimeters of a 70% solution (by weight) of sulfuric acid at 100° F. (38° C.) and work it for 15 minutes. Filter on a Gooch filter or 100-mesh screen and wash well with cold water. Then place the sample in a beaker of 2% sodium bicarbonate at room temperature for 5 minutes. Filter again, wash well on the filter, dry and weigh. This method dissolves all the ordinary fibers except wool which is left as a residue. No correction factor is necessary for a normal wool, but reworked and re-used wools require a correction factor. When nylon is one of the fibers dissolved in this method, the first washing after the 70% acid may cause precipitation of the nylon on the wool. To avoid this, the wool residue should first be washed with 70% sulfuric acid and then with water. Wool-casein mixture cannot be analyzed by this method. The 10% potassium hydroxide method must be used.

Choosing the Method

Table XXIII gives the solubility of the various fibers by the above methods. It will be noted that in several cases in Table XXIII are the words *partly soluble.* In these cases, the method cannot be used if the fibers in question are present because the fibers dissolve to a considerable extent but cannot be corrected because of their extreme variation. Thus, if silk, viscose, and cotton are present, we cannot first dissolve the silk with cold concentrated hydrochloric acid because the viscose dissolves to an unknown extent. In this case, the potassium hydroxide method would be used for silk, followed by the calcium thiocyanate method for viscose. The fibers given in capitals are the principal fibers met with, those beneath in small letters are the less common fibers to which the correction factors will *probably* apply.

The methods to use will depend upon what fiber mixture we have. In some cases, there will be a choice of methods, the choice will be governed by the accuracy desired or the convenience or speed of the method. The following mixtures of two fibers may arise:

1. Acetate rayon and any other fiber. Use the acetone method.

2. Cotton and wool. Use the sulfuric acid method for the most accuracy, or the potassium hydroxide method for less critical work.

3. Cotton and silk. Use the potassium hydroxide method, the

hydrochloric acid method (less accurate), or the calcium thiocyanate method (expensive reagent, longer time).

4. Cotton and viscose. Use the calcium thiocyanate method.

5. Viscose and wool. Use the potassium hydroxide method (less accurate), the calcium thiocyanate method, or the sulfuric acid method (best).

6. Viscose and silk. Use the potassium hydroxide method.

7. Silk and wool. Use the hydrochloric acid method (most convenient), the calcium thiocyanate method, or the sulfuric acid method.

8. Nylon and other fibers. Few mixtures of nylon and other fibers have appeared as yet. Mixtures with cotton may be analyzed by the acetic acid method; with silk by the thiocyanate or potassium hydroxide method; with wool by the sulfuric acid method, hydrochloric acid method, or acetic acid method; with acetate rayon by the acetone method; with viscose rayon by the thiocyanate or acetic acid method.

In analyzing mixtures of three or four fibers, we may remove one fiber, weigh, remove another fiber and weigh again; another and more accurate method is to take two samples and make a different determination on each; this last method avoids the use of more than one reagent on any residue and thus does not create an unknown correction factor. The second method is also quicker, since only two dryings are necessary instead of three or four. For example, a mixture of cotton, wool, and silk could be analyzed on one sample by treating with hydrochloric acid and weighing, then treating with potassium hydroxide and weighing again, but the acid treatment might alter the cotton so that the correction factor for the potassium hydroxide residue would not be 3% but some higher figure. A more accurate way would be to determine wool on one sample (by the sulfuric acid method) and cotton on another sample (by the potassium hydroxide method) and determine the silk by difference. By the use of Table XXIII, the various combinations of methods to use for various combinations of fibers may be determined. Of course, acetone does not affect any of the fibers except acetate rayon, so a mixture of acetate and any other number of fibers is first treated with acetone to determine the acetate rayon and is then treated with other reagents

Use of Correction Factors

The correction factor is the figure by which a weight of a given residue must be multiplied to give the true weight of the residue before the chemical treatment took place. If we let

S = solubility given in Table XXIII
C = correction factor

then

$$C = \frac{100}{100 - S} \qquad \text{or} \qquad C = \frac{1}{1 - \frac{S}{100}}$$

Thus the correction factor for cotton, after the potassium hydroxide treatment, is $100/100 - 3 = \frac{100}{97}$ or $1/0.97$. Wool, after the sulfuric acid treatment, requires no correction, and none of the fibers requires correction after the acetone process. The use of the correction factors may best be shown by examples:

1. Mixture = cotton and wool
   Method used = potassium hydroxide
   Residue = cotton
   Correction factor = $100/100 - 3 = \frac{100}{97}$
   Weight of sample = 5.14 grams
   Weight of residue = 2.39 grams
   True weight of cotton = $2.39 \times \frac{100}{97} = 2.46$ grams
   Per cent cotton = $2.46 \times 100/5.14 = 47.9\%$
   Per cent wool = $100 - 47.9 = 52.1\%$
2. Mixture = cotton, wool, silk
   Method used = hydrochloric acid method followed by potassium hydroxide method
   Weight of sample = 4.98 grams
   Weight residue after HCl = 3.05 grams
   Weight residue after KOH = 2.13 grams
   Weight of cotton before KOH = $2.13 \times \frac{100}{97} = 2.20$ grams

Weight of wool before KOH = 3.05 − 2.20 = 0.85 grams
Weight of cotton before HCl = 2.20 × $\frac{100}{96}$ = 2.29 grams
Weight of wool before HCl = .85 × 100/99.5 = 0.85 grams
Weight of silk before HCl = 4.98 − 2.29 − .85 = 1.84 grams
Per cent cotton = 2.29 × 100/4.98 = 46.0%
Per cent wool = 0.85 × 100/4.98 = 17.1%
Per cent silk = 1.84 × 100/4.98 = 36.9%

3. Mixture = silk, wool, viscose
Methods used = sulfuric acid (sample A) and potassium hydroxide (sample B)

A

Weight of sample = 2.0575 grams
Weight residue = 0.5500 grams
True weight of wool = 0.5500 grams

$$\text{Per cent wool} = \frac{0.5500}{2.0575} \times 100 = 26.8$$

B

Weight of sample = 4.9590 grams
Weight residue = 2.4865 grams
True weight of viscose = 2.4865 × $\frac{100}{95}$ = 2.62 grams

$$\text{Per cent viscose} = \frac{2.62}{4.96} \times 100 = 52.8$$

Per cent silk = 100 − (26.8×52.8)
= 100 − 79.6
= 20.4

Special Cases

In addition to the cases which have been discussed, there are certain special cases which deserve mention:

1. Mixtures of fibers in the same group in Table XXIII. For example, mixtures of wool and mohair or cotton and linen. These mixtures cannot be analyzed by chemical means but must be picked apart mechanically if possible or analyzed by

means of the microscope. This will be discussed later in the section on microscopic analysis.

2. Mixtures of fibers of which one fiber is not normal but has been degenerated by some previous treatment. For example, mixtures of shoddy (somewhat degraded wool) and cotton. These are best analyzed by a method which dissolves the degenerated fiber and leaves the normal fiber as a residue.

3. Felts. These are so compactly packed that certain removal of relatively small amounts of cotton is nearly impossible. On the other hand, if the larger amount of wool is removed the cotton does not hinder the operation so much. The A.S.T.M. standard method for felts is the same as the potassium hydroxide method except that 5% sodium hydroxide is used, and the solubility of the cotton is then 5% (correction factor = $\frac{100}{95}$).

4. Mixtures of asbestos and organic fibers. These are analyzed by taking a five-gram sample which is ignited in an electric furnace at 800 to 810° C., after which the residue (asbestos) is weighed. The asbestos loses about 14% in weight as a result of losing the water of crystallization, so that the correction factor of $\frac{100}{86}$ is used.

5. The A.S.T.M. suggests the separation of wool-casein mixtures by use of a liquid of 1.310 ± .005 specific gravity. In this liquid, the casein would float and the wool would sink. The liquid may be prepared by mixing 5 volumes of carbon tetrachloride and 3 volumes of toluene.

6. McKay has worked out a method of determining the amount of viscose (but not cupra rayon) in a mixture with cotton, wool, or silk, which uses cold dilute sodium hydroxide. A freezing solution at $-3°$ C. is made by placing 1200 grams of ice, 500 cubic centimeters of water, and 80 grams of common salt in an insulated container. A sample, weighing 0.2 gram, is taken for analysis, and another sample is taken for regain. Into each of a number of test tubes is placed 50 cubic centimeters of 2.0 ± 0.1N sodium hydroxide. The test tubes and the sample are cooled to $-3°$ C. ± 2°. After $\frac{1}{2}$ hour, the cold alkali is poured on the sample and allowed to stand at $-3°$ C. for 1 hour,

with shaking every 10 minutes. The sample is filtered by suction, rinsed with 25 cubic centimeters of 2.0N sodium hydroxide at −3° C., then with 50 cubic centimeters of distilled water. The sample is then rinsed with 200 cubic centimeters of 0.2N hydrochloric acid, washed, dried, and weighed. The solubility of cotton and wool is 0.5% and of silk is 0.0%.

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Chapter 15

SWELLING AND DAMAGE IN CELLULOSE FIBERS

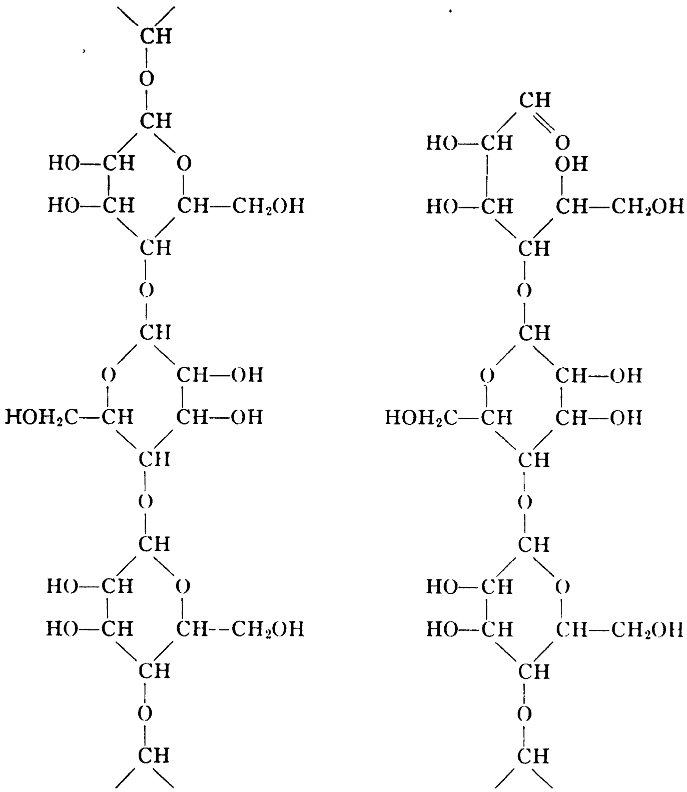
Introduction

Before studying the specific tests for the condition of a cellulose fiber, it would be well to consider briefly the structure and the chemistry of cellulose and its derivatives. Cellulose, itself, as shown in a, Fig. 50, consists of a long chain of glucose anhydride units held together lengthwise by an oxygen linkage and sideways by partial valence. The cellulose chain terminates in a glucose residue which contains an aldehyde group (b, Fig. 50) and gives cellulose a very slight reducing action. The chemical activity of cellulose centers around the hydroxyl groups and the oxygen linkages.

Swollen cellulose may be produced by one or more of several methods:

1. Strong acids such as concentrated sulfuric acid
   Examples: amyloid, artificial parchment, Swiss finish, Heberlein finish
2. Strong alkalies such as sodium hydroxide and cuprammonium hydroxide
   Examples: mercerized cotton, cupra rayon
3. Certain salts such as calcium thiocyanate and zinc chloride
4. Esterification followed by solution in a solvent and reprecipitation of the cellulose.
   Examples: acetate rayon, viscose rayon

The swollen cellulose has the same structural formula as ordinary cellulose, the only difference (apart from incidental degradation) being that the partial valence forces are destroyed or decreased so that the chains are not as well oriented and the



a.
Middle of cellulose molecule

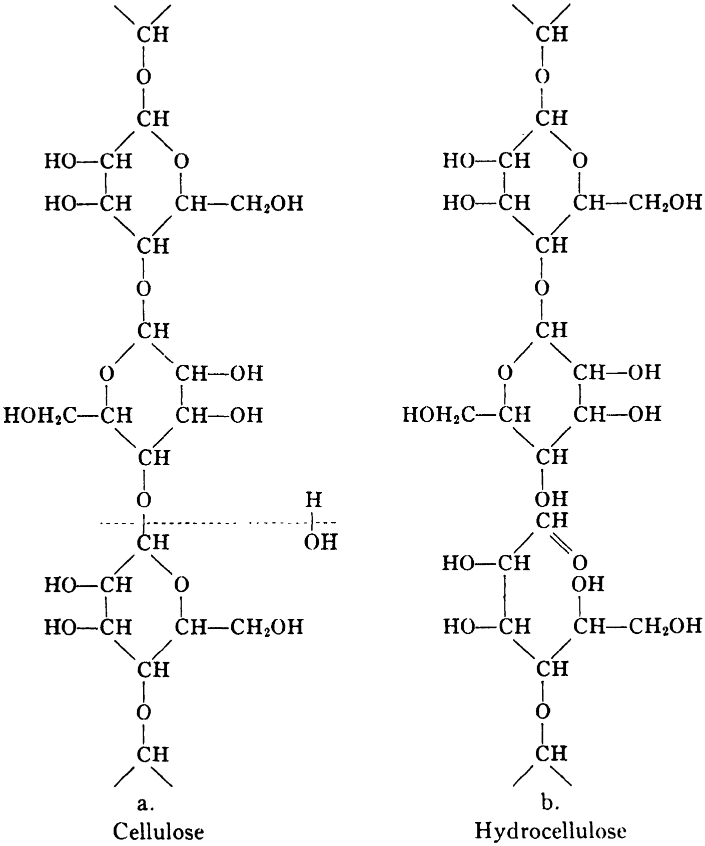
b.
End of cellulose molecule

FIG. 50. Structural Formula of Cellulose

hydroxyl groups are more available for reaction. We find then that swollen cellulose has the same reactions as cellulose but that the reactions proceed further or proceed more rapidly. This may be shown in several ways:

1. Faster chemical reactions
    - a. Hydrolysis by acids
    - b. Oxidation, e.g., by sodium hypobromite
    - c. Esterification, e.g., acetylation

2. Increased absorption
   a. Moisture (see Table IV for regains)
   b. Direct dyes, e.g., benzopurpurin
   c. Iodine, e.g., zinc chloride-iodine reagent
   d. Alkalies, e.g., barium hydroxide


a. Cellulose

b. Hydrocellulose

FIG. 51. Formation of Hydrocellulose

Of these last two phenomena, the absorption of iodine from zinc chloride-iodine furnishes us with a qualitative test for swelling, and the absorption of barium hydroxide furnishes us with a quantitative test for mercerization.

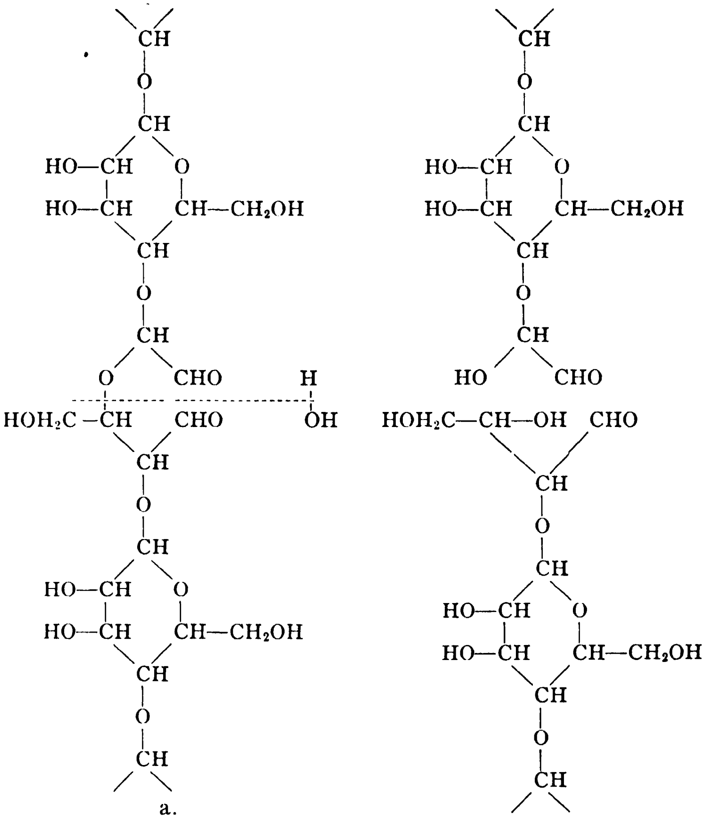
TABLE XXIV
COMPARISON OF TESTS ON CELLULOSE DERIVATIVES

	Cellulose		*Hydrocellulose*		*Oxycellulose*	
			From Acid	*From Acid*	*Alkaline*	*Acid*
Test	*Swollen*	*Unswollen*	*Solution*	*Dried-in*	*Oxidation*	*Oxidation*
Zinc chloride-iodine	Blue	White	Blue	Blue	Blue	Blue
Fehling's solution	White	White	Red	Red	Light red	Red
Turnbull's blue	White	White	White	White	Blue	Light blue
Loss in sodium hydroxide	Low	Low	High	High	High	High
Methylene blue absorption	Low	Low	Low	High	High	Medium
Copper number	Low	Low	High	High	Medium	High
Fluidity in cuprammonia	Low	Low	High	High	High	High

The degradation of cellulose is accomplished by hydrolysis of the oxygen linkage which results in shorter molecules and is indicated by a higher fluidity (or lower viscosity) and an increased solubility in dilute alkali. The degradation is also accomplished by an oxidation of the hydroxyl groups to aldehyde groups, indicated by an increased ability to reduce alkaline copper salts, and further oxidation to carboxyl groups, indicated by an increased ability to absorb basic dyes as methylene blue or to form ferrous salts as in the Turnbull's Blue test. Degradation by oxidation is usually, also, accompanied by hydrolysis.

Hydrocellulose is produced by the action of acids on cellulose, as shown in Fig. 51, to give molecules similar to cellulose but shorter, and since more ends of the chains now exist, the reducing power (due to the aldehyde groups) is larger. Two forms of hydrocellulose may be obtained: one from the treatment of cellulose by hot dilute acids (acid solution type); and one from the impregnation of cellulose with sulfuric or phosphoric acid, followed by drying of the material with consequent concentration of the acid (acid dried-in type). The general chemical properties of these two types of hydrocellulose are shown in Table XXIV.

Oxycellulose is produced by the action of oxidizing agents on cellulose. If the oxidation is carried out in acid solution, the product differs from cellulose in containing a large number of aldehyde groups (a, Fig. 52) and a relatively small number of carboxyl groups. The chain is not broken until a treatment with alkali is given (b, Fig. 52) as in boiling out and laundering. The strong alkali used in the fluidity test also breaks the chain and causes high fluidities. If the oxidation is carried out in alkaline solution, the number of aldehyde groups is reduced and the number of carboxyl groups increased by oxidation, and the alkaline medium also causes breaking of the molecular chain (a, b, and c, Fig. 53). The general chemical properties of these two types of oxycellulose are shown in Table XXIV.



a.
Oxycellulose formed
by acid
oxidation

b.
Oxycellulose from
acid oxidation, after
alkali treatment

FIG. 52. Oxycellulose Obtained by Oxidation in Acid Solution

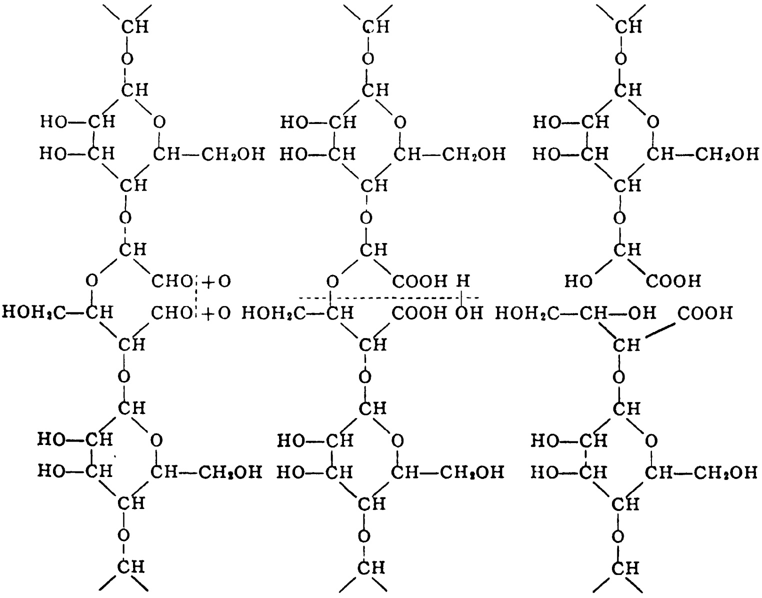
The tests which will be considered in this chapter may be classified as follows.

A. Qualitative tests
  1. For swollen cellulose
    a. Zinc chloride-iodine test (see Chapter 14).

2. For chemical damage
    - a. Fehling's solution for hydrocellulose or oxycellulose
    - b. Turnbull's blue for oxycellulose

B. Quantitative tests
1. For swollen cellulose
    - a. Barium activity number for mercerized cotton
2. For degraded cellulose
    - a. Loss of weight in hot dilute NaOH for molecule length
    - b. Methylene blue absorption for carboxyl groups
    - c. Copper number for aldehyde groups
    - d. Fluidity for molecule length

The first part of Table XXV gives the results of the above quantitative determinations for purified cellulose and for the



a. First stage b. Second stage c. Third stage

FIG. 53. Oxycellulose Obtained by Oxidation in Alkaline Solution

Table XXV

QUANTITATIVE DATA ON CELLULOSE DERIVATIVES

	Loss in Sodium Hydroxide in One Hour	*Methylene Blue Absorption at pH7*	*Copper Number*	*Fluidity in 0.5% Solution*
Bleached cotton linters or purified cotton	not over 1.5	0.8–0.9	less than 0.1	1–2
Viscose rayon	6–11	1.3–2.3	0.8–1.5	8–14*
Cupra rayon	6–11	1.3–2.2	0.5–1.5	2.5–6*
Acetate rayon	45–48	0.8–1.2	2.8–3.2	53–54*
Cotton damaged by acid:				
Loss in strength = 10%	—	—	0.3	10
Loss in strength = 30%	—	—	0.8	18
Loss in strength = 50%	—	—	1.4	27
Cotton damaged by acid oxidation:				
Loss in strength = 10%	—	—	0.5	10
Loss in strength = 30%	—	—	2.0	20
Loss in strength = 50%	—	—	4.2	27
Cotton damaged by alkaline oxidation:				
Loss in strength = 10%	—	—	0.2	10
Loss in strength = 30%	—	—	0.5	20
Loss in strength = 50%	—	—	1.8	27

* In 2% solution.

various rayons. The last part of Table XXV gives the approximate copper numbers and fluidities which may be expected in cotton goods which have been damaged in various ways so as to produce losses in strength of 10%, 30%, and 50%. In commercial practice, a damage resulting in not over 10% loss in strength is permissible if done to produce a certain result. For example, in bleaching, a loss in strength is almost inevitable but should not exceed 10%. The 30% and 50% values are included to give an idea of what values are obtained in the case of excessively damaged materials.

Qualitative Tests

ZINC CHLORIDE-IODINE TEST

The zinc chloride-iodine test for swollen cellulose is given in Chapter 14. The blue color obtained by this test is not permanent. If it is desired to keep a record of the color, the sample after removal from the reagent and rinsing is dropped into a boiling solution of indigosol black 1B or indigosol 04B (the concentration is immaterial). The iodine causes an immediate dyeing of the sample which is then washed first in cold water and then in boiling soap solution, after which it may be kept for reference. If the sample to be tested is dyed, the color may be stripped by any of the ordinary methods without influencing this test.

USE OF FEHLING'S SOLUTION

Fehling's solution, the formula of which is given in Chapter 13, may be used as a test for damage since both hydrocellulose and oxycellulose contain aldehyde groups which cause a reduction of alkaline copper salts to insoluble red cuprous oxide. The test is carried out as follows: Mix 10 cubic centimeters of solution A with 10 cubic centimeters of solution B and heat to boiling. Add the sample and boil for several minutes. Remove and rinse well. The presence of red or pink spots shows chemical damage (hydrocellulose or oxycellulose). This test, as we shall see later, may be made quantitative (with some modifications) and gives us the *copper number.*

TURNBULL'S BLUE TEST

The Turnbull's blue test depends upon the fact that, when immersed in ferrous sulfate, oxycellulose, because of its carboxyl groups, will form a ferrous salt which will not wash out, whereas cellulose and hydrocellulose, which do not contain carboxyl groups, will not hold the ferrous salt. If then the sample is placed in potassium ferricyanide, the ferrous salt of oxycellulose will give a blue color (Turnbull's blue), whereas cellulose and hydrocellulose will not give any color. The test is carried out as follows: Immerse the sample in a 1% solution of ferrous sulfate at room temperature for 10 minutes. Then wash thoroughly in running water; immerse the sample in a 1% solution of potassium ferricyanide at room temperature for 5 minutes. Remove and wash. Cellulose will be tinted very faintly blue, hydrocellulose still more faintly, but oxycellulose will be a deep blue. If the sample is dyed, the color may be stripped by either of the following methods, which do not affect the results of the test unduly.

1. Use a 0.5% solution of hydrosulfite and a little ammonia at 40° C. for 30 minutes.
2. Use a 0.1% solution of titanous sulfate or chloride at 40° C. for 30 minutes.

OTHER TESTS

Other qualitative tests given in the literature have been tried by the author and found to be not as reliable or at any rate no more reliable than the above and rather more complicated. These tests include:

Methylene blue test
Diamine sky blue test
Benzopurpurin test
Indanthrene Yellow test
Harrison's test (alkaline silver salt)
Heat test

Quantitative Tests

BARIUM ACTIVITY NUMBER

The increased absorption of swollen cellulose for various substances is most conveniently and accurately measured in the case of barium hydroxide. The original method of Neale has been greatly simplified by Edelstein who also found that the results are unaffected by the presence of dye.

Solutions used:

Barium hydroxide solution, approximately 0.25N (may vary between 0.16N and 0.30N).

Standardized 0.1N hydrochloric acid.

The sample should be freed from oils, fats, waxes, etc., by extraction, and finishing materials should be removed by scouring. A sample of unmercerized American cotton should be run at the same time. The samples are weighed out at room conditions and no regain correction is necessary. Two-gram samples are weighed out exactly on a centigram balance and placed in small conical flasks. Using a ten-cubic centimeter pipet, 30 cubic centimeters of barium hydroxide solution are pipetted into each flask. The flasks are stoppered and allowed to stand at room temperature with frequent shaking for at least 2 hours. After 2 hours, 10 cubic centimeters of barium hydroxide are withdrawn from each flask and titrated with the standard hydrochloric acid using phenolphthalein indicator. A blank titration is also run on 10 cubic centimeters of unused barium hydroxide. The same pipet and buret should be used throughout the test. The barium activity number is defined as:

$$\frac{\text{Barium hydroxide absorbed by mercerized sample}}{\text{Barium hydroxide absorbed by unmercerized sample}} \times 100$$

and since the normality of the barium hydroxide is the same and the weight of sample is the same,

$$\text{Barium activity number} = \frac{a - b}{a - c} \times 100$$

where

a = cubic centimeters of acid for blank
b = cubic centimeters of acid for mercerized sample
c = cubic centimeters of acid for unmercerized sample

Unmercerized American cotton, of course, is 100 on this scale and a remarkable uniformity is obtained on all samples tested, so that any American cotton may be used as the standard. A large number of commercially mercerized cotton cloths gave values of 116–130, and a number of commercially mercerized cotton yarns gave values of 150–160. If the above mercerized cloths are remercerized in the laboratory, their barium activity numbers may be raised to 150–160 also.

SOLUBILITY IN SODIUM HYDROXIDE

In the absence of any finishing compounds, this test is a very useful one since it is simple to carry out and applicable to any type of damage. Two methods have been suggested: the 10N-2N method, which is rather complex, and the N/4 method, which is the simplest method and the one most commonly used. This method will be given here. The original method called for a four-hour boil, but this is almost universally reduced to 1 hour.

A two-gram sample is dried and weighed and is then boiled for 1 hour in 200 cubic centimeters of N/4 sodium hydroxide (1% solution), using a reflux. The liquor is decanted into a Gooch crucible, the residue is washed with sodium hydroxide solution, then with water, and finally with N/10 sulfuric acid. The residue is collected in the crucible and washed with water until the washings are neutral to litmus. The crucible and contents are dried for 3 hours at 110° C. and weighed.

Using this method, the loss in weight of a normal cellulose is 1.5% and anything over this value is indicative of damage. It has also been shown that, for any kind of damage, the loss in sodium hydroxide is six times the copper number up to a copper number of 2.5; beyond this point hydrocelluloses lose more and oxycelluloses lose less than six times the copper number.

METHYLENE BLUE ABSORPTION

The absorption of methylene blue by cellulose derivatives is generally due to the carboxyl groups present in oxycelluloses formed by alkaline oxidation, but it may also be due to mineral acids which have been dried into the cellulose and are not easily washed out (hydrocelluloses of the acid dried-in type). A distinction may be made between these two, however, by previously boiling in dilute sodium hydroxide which does not materially change the methylene blue absorption of the oxycellulose but decreases it in the case of the hydrocellulose.

The method depends upon treating the sample with a methylene blue solution of known strength and buffered to a known pH (usually a pH of 7) and determining the concentration of the methylene blue in the solution either by titration with naphthol yellow S or by the colorimetric method. A buffered solution is necessary because the methylene blue is very sensitive to traces of acid and alkali; even the alkali from the laboratory glassware affects the results if the solution is unbuffered.

Purification of Methylene Blue

The ordinary methylene blue hydrochloride (molecular weight = 320) is recrystallized three times from twelve times its weight of water. The moisture is then determined on a small portion of the powder, and the remainder is used to make up the solution. In accurate work, the purity of the methylene blue hydrochloride may be determined by titanous chloride.

Preparation of Solutions

An M/250 solution of methylene blue at pH 7 is prepared by mixing 6.81 grams of potassium dihydrogen phosphate, 29.63 cubic centimeters of N sodium hydroxide, and 1.279 grams of pure methylene blue hydrochloride and making up to 1 liter with water. An M/250 solution of methylene blue at pH 2.7 may be prepared by taking 1.279 grams of pure methylene blue hydrochloride and making up to 1 liter with N/5 acetic acid.

The naphthol yellow S solution should contain 1.5 to 2.0 millimoles per liter and should be standardized against the methylene blue. The addition of the naphthol yellow S to the methylene blue solution produces a brown precipitate, and the solution becomes less and less blue and finally changes to yellow. The end-point may best be detected by observing a drop suspended on a glass rod, or by spotting on filter paper, or better by centrifuging.

The titration method is carried out as follows: A sample weighing 2.5 grams is placed in a test tube or small conical flask and 15 cubic centimeters of methylene blue are added from a buret; the sample is well worked in the solution and is left for 18 hours at room temperature. With a pipet, 10 cubic centimeters of solution are withdrawn and titrated with the naphthol yellow S. The methylene blue absorbed by the sample is calculated in millimoles per 100 grams of dry sample (moisture determined on a separate sample).

The colorimetric method is carried out as follows: Use M/2500 methylene blue solution obtained by diluting the M/250 solution to one-tenth of its original strength. From 1.5 to 2.0 grams of sample in small pieces are shaken with 50 cubic centimeters of methylene blue solution at room temperature for 18 hours. Some of the solution is then removed and compared in a colorimeter with methylene blue solution of a concentration of M/5000. The results are calculated as in the titration method.

If the values at pH 7 and pH 2.7 are to be determined, the test may be carried out at pH 7. The sample may be washed with water, then treated at the boil with N/5 acetic acid until only a slight blue color is left. It is then washed and used for a test at pH 2.7.

Uses of the Methylene Blue Absorption

1. To distinguish various cottons. At pH 7, American cottons have values of 0.8 to 0.9, Egyptian cottons 1.0 to 1.1, and Sea Island cottons about 1.0.
2. To distinguish between oxycelluloses due to acid oxidation

(low methylene blue absorption) and alkaline oxidation (high methylene blue absorption). See Table XXII.

3. To distinguish the acid dried-in type of hydrocellulose. In the case of cellulose or oxycellulose, the absorption at pH 7 is greater than at pH 2.7, whereas in the case of hydrocellulose of the acid dried-in type, the absorption at pH 7 is less than at pH 2.7. This identification may also be made by a previous treatment with dilute sodium hydroxide, as mentioned earlier in this section.

The disadvantages of the methylene blue absorption are:

1. The colorimetric method requires a colorimeter; the titration method requires a centrifuge for a clear end-point. These pieces of apparatus are expensive and not always available.
2. The test requires over 18 hours to carry out.
3. The results are of value chiefly for differentiation. Other tests give as good a measure of damage and require less time and equipment.

COPPER NUMBER

Copper number may be defined as the number of grams of copper reduced from the cupric to the cuprous state in alkaline solution by 100 grams of dry sample. The reduction, in the absence of any sugars or other extraneous material, is due to the aldehyde groups present and consequently approaches zero for pure cellulose but is quite high for hydrocellulose and oxycellulose. The copper number was originally carried out as a quantitative evaluation of the Fehling's solution test which has been described as a qualitative test for damage to cellulose, but it was found that the strong alkali used in Fehling's solution in the presence of the oxygen of the air caused a progressive oxycellulose formation. For this reason, a modified solution is employed in the now universally accepted Schwalbe-Braidy method which will be given in detail.

Solutions used:

Solution A: 100 grams of crystalline copper sulfate per liter

Solution B: 50 grams of sodium bicarbonate and 350 grams of sodium carbonate ($Na_2CO_3.10H_2O$) per liter

Ferric alum: 100 grams of ferric alum and 140 cubic centimeters of concentrated sulfuric acid per liter

Dilute sodium carbonate: 5 grams of sodium carbonate (Na_2CO_3) per liter

Dilute sulfuric acid: approximately 2N

Standard permanganate: N/25 potassium permanganate (standardized)

Procedure: Determine regain on a separate sample. Over a sample weighing 2.5 grams in a conical flask having a capacity of 100 cubic centimeters is poured a boiling mixture of 5 cubic centimeters of solution A and 95 cubic centimeters of solution B. The mouth of the flask is covered, and the flask is placed in (not on top of) a rapidly boiling water bath or in a steam bath for exactly 3 hours. The contents are then filtered by suction through a fritted glass filter or Gooch crucible with asbestos mat, and the residue is washed first with dilute sodium carbonate and then with water. The filtrate and washings are discarded. Over the residue on the filter is poured 50 cubic centimeters of ferric alum solution divided into four lots, and the residue is then washed with 50 cubic centimeters of dilute sulfuric acid. The combined ferric alum and acid washings are titrated with the standard permanganate. A blank is also run on 50 cubic centimeters of unused ferric alum and 50 cubic centimeters of acid. A sample determination follows:

a. Weight of bottle and sample	41.3650 grams
b. Weight of bottle and dry sample	41.2735 grams
c. Weight of bottle alone	37.7655 grams
d. Weight of moisture (a − b)	0.0915 grams
e. Weight of dry sample (b − c)	3.5080 grams
f. Regain $\left(100\,\frac{d}{e}\right)$	2.60%

g. Weight of sample	2.52 grams	2.49 grams
h. Dry wt. of sample $\left(\frac{g}{1+\frac{f}{100}}\right)$	2.46 grams	2.43 grams
i. Volume of $KMnO_4$ used	9.55 cubic centimeters	9.30 cubic centimeters
j. Volume of $KMnO_4$ for blank	0.15 cubic centimeters	0.15 cubic centimeters
k. True volume of $KMnO_4$ (i − j)	9.40 cubic centimeters	9.15 cubic centimeters
m. Normality of $KMnO_4$	0.0395	0.0395
n. Milliequivalent weight of copper	0.0636	0.0636
o. Copper number $\left(100 \times k \times m \times \frac{n}{h}\right)$	0.96	0.94
p. Copper number (average)	0.95	

A blank of 0.01 may be obtained by running a test with no cellulose present, and theoretically this should be subtracted from any value found, but actually it is generally neglected. The method is fairly critical so that the time, temperature, and weight of sample must be as specified. The amount of copper present in the solution is enough to give a copper number of 5.2 as a maximum, so that if a first determination shows a copper number of 4 or over, the determination should be repeated, using a one-gram sample, instead of a sample weighing 2.5 grams, in which case copper numbers up to 10 may be determined.

A micro-method for use on very small samples is also available: a sample weighing 0.25 gram is taken in a 4 × $\frac{3}{4}$ inches covered test tube. To 9.5 cubic centimeters of B is added 0.5 cubic centimeter of A. The mixture is heated to boiling and poured over the sample. The test tube is immersed in boiling water for 3 hours, with stirring after 10 minutes to remove carbon dioxide. The mixture is filtered on a fritted glass filter or Gooch crucible and asbestos mat and washed with distilled water three times. Then 5 cubic centimeters of ferric alum are added to the reaction tube and poured over the sample on the filter in several lots. The residue is washed with several lots of distilled water of 2 cubic centimeters each. The ferric alum and washings are then titrated with standard permanganate, using a microburet reading to 0.005 cubic centimeter. A blank is run on 5 cubic centimeters of ferric alum solution.

Bleached cotton will range from 0.01 to 0.3. Cottons which have had a thorough scouring and a mild bleach have the lower range of values (0.01 to 0.1), and cottons which have had a less thorough scouring and a more vigorous bleach have the upper range (0.1 to 0.3). The maximum value compatible with good bleaching is 0.3, and preferably a good bleached cotton should be below 0.1. Oxycelluloses formed by oxidation to the point of disintegration of the fiber will range from 6 to 40, and hydrocellulose formed by hydrolysis to disintegration will range from 3.5 to 9.0. The values to be expected for normal rayons and cotton damaged to various extents are given in Table XXV.

The copper number, and the solubility in dilute sodium hydroxide are related, as we have already seen in the discussion of the solubility test.

Copper number is, in general, a useful test for damage. It has only two disadvantages:

1. It is not sensitive to incipient damage. As shown in Table XXV, up to 10% damage may exist with very little change in copper number.

2. A sample which has been given an alkaline boil-off (scouring or laundering) after being damaged but before being tested for copper number will not show damage by the copper-number test unless the damage is very great.

FLUIDITY

Viscosity is the internal friction of a liquid on itself, or its resistance to flow; its units are dynes per square centimeter per centimeter per second or poises. Fluidity is the ease of flow and is the reciprocal of viscosity; its units are reciprocal poises:

$$F = \frac{1}{V}$$

where

V = viscosity in poises

F = fluidity in reciprocal poises or rhes.

There are three general methods of measuring viscosity or fluidity:

1. Resistance to flow through a capillary tube
2. Resistance to fall of a ball through the fluid
3. Resistance to rotation of a disc or cylinder in the fluid

An instrument which measures the viscosity is known as a viscometer. We have three general types of viscometers based on the three methods above, but the capillary-flow type of viscometer is best adapted to liquids of low viscosity (high fluidity) and is the only one which we shall consider at this time. In the determination of the fluidity of cellulose and its derivatives, the Clibbens and Geake method and viscometer is most commonly used and will be discussed.

It has been shown that cuprammonium hydroxide solution (to be referred to hereafter as *cuprammonia*) will react with and dissolve cellulose and its derivatives without breaking the molecular chain except in the case of one oxycellulose, as mentioned in the introduction of this chapter; cuprammonia, therefore, is used as the solvent for the cellulose. Since the solution of the cellulose presents some difficulties, a standard composition and method of preparation of the solvent has been worked out. The solvent should contain:

15 ± 0.1 gram of copper per liter
200 ± 5 grams of ammonia per liter

and should have the following physical properties at 20° C.:

$d = 0.94$ where d = density in grams per cubic centimeter
$F = 72$ where F = fluidity in reciprocal poises.

To prepare this solvent, the following procedure should be used: Mix 2.6 liters of concentrated ammonia (density = 0.88), 0.4 liters of water, and 3 grams of cane sugar. Stir the solution mechanically at 400 revolutions per minute with 180 grams of precipitated copper which has passed a 60-mesh sieve. Blow through air at the rate of 10 liters per hour. The air should have been previously bubbled through strong ammonia. This reaction is allowed to proceed for 5 hours, after which the solution is analyzed for copper and ammonia content and adjusted to the standard concentrations. The final solvent should be stored in an inert atmosphere or kept in full containers which are just large enough to hold one viscometer charge or a little over. Copper is determined colorimetrically by comparison with solutions of known copper content, and the ammonia is determined by titrating a portion of the solvent with standard acid and subtracting the amount of acid corresponding to the already determined copper content.

The Clibbens and Geake viscometer is shown in Fig. 54 and should be made as closely as possible to the standard dimensions. In this country, the viscometer and its support may be obtained from laboratory supply houses under the name of *pulp viscom-*

eter. The time at 20° C. is measured for the level of liquid to fall from the top ring to the bottom ring. The middle ring may be used to check on the results, since for a truly viscous liquid the time for the level to fall from the top ring to the middle ring should equal the time from the middle to the bottom ring. The viscometer should be fitted at the bottom with a short piece of rubber tubing and a clamp and at the top with a stopper and capillary tube with rubber tubing and clamp attached.

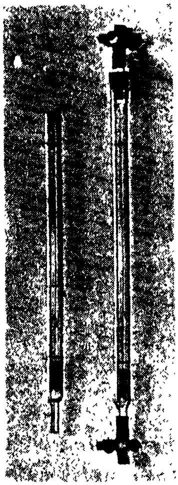


FIG. 54. Cuprammonia Viscometer

The instrument should be calibrated by means of a liquid of known fluidity and density which flows slowly enough to cause very little error by kinetic-energy effects. Glycerin solutions are of such a nature. If we let

F = fluidity in reciprocal poises at 20° C.
d = density in grams per cubic centimeter at 20° C.
t = time of flow in seconds
K = the constant of the instrument

Then

$$F = \frac{K}{dt} \quad \text{or} \quad K = Fdt$$

and if we take a glycerin solution of such composition that

$$d = 1.1681$$

and

$$F = 6.83$$

then

$$K = 1.1681 \times 6.83t = 7.98t$$

Theoretically this should receive a small correction for kinetic energy, but this is usually neglected. The value of K, if the instrument is made according to specifications, should be roughly 2000. The total volume (V) of the viscometer in cubic centimeters with the clamps in place should also be obtained by weighing empty and then weighing full of the distilled water; this volume will be roughly 20 cubic centimeters. The values of K and V for each instrument should be recorded in the laboratory log-book.

The cellulose sample is dissolved in the viscometer in which its fluidity is to be determined, using a 0.5% solution for cottons and a 2% solution for rayons. Since it is difficult to get exact weights of dry sample, we usually make the solutions approximately of this strength and afterward correct the results to the proper concentration. The sample, about 0.1 gram of cotton or 0.4 gram of rayon, is weighed out, and at the same time another sample is weighed out for the regain determination. A small amount of cuprammonia solution is placed in the viscometer, the bottom clamp opened to fill the capillary, the sample (preferably cut up into short lengths) is added, and 0.7 cubic centimeter of mercury run in from a buret (to stir the solution). (Instead of mercury, the A.S.T.M. specifies a wedge-shaped steel plunger with a volume of 0.7 cubic centimeter and a diameter of 7 millimeters.) The viscometer is filled to the top, and the stopper added; the excess cuprammonia runs out of the top capillary which is then closed with the clamp. The viscometer is then wrapped in a black cloth to prevent light damage and placed on a wheel which revolves at four revolutions per minute until the cellulose dissolves. This is usually overnight.

When the cellulose is all dissolved, the bottom tubing and clamp are removed, the viscometer is placed in its support which, in turn, is in a constant-temperature bath at 20° C., and allowed to come to constant temperature. When constant temperature is arrived at, the top clamp is opened, and the mercury runs out, followed by the cellulose solution. The time for the level to fall from the top ring to the bottom one is measured with a stop watch. The fluidity is then calculated from the formula:

$$F = \frac{K}{dt}$$

where d may be taken as 0.94 and K has already been determined, so that

$$F = \frac{K}{0.94t}$$

Theoretically, this should have a correction for kinetic energy, but this is not usually done because it is negligible in commercial work. The concentration of the solution in per cent is calculated from the dry weight of the sample and the volume of the viscometer

$$C = \frac{100W}{V - 0.7}$$

where

C = concentration in per cent (weight/volume)
W = dry weight of sample taken in grams
V = volume of viscometer in cubic centimeters
0.7 = volume of mercury in cubic centimeters

To correct the results to the standard concentration, we use the formula:

$$1 + \frac{A}{C} = \frac{B}{\log \frac{F_s}{F}}$$

where

F_s = fluidity of the pure cuprammonia solvent = 72 poises
B = a constant = 11

and

A = *concentration correction constant*

Then

$$A = C\left(\frac{11}{\log\frac{72}{F}} - 1\right)$$

Substituting the value of A just found in the formula and letting C = 0.5 or 2.0, according to whether cotton or rayon is being tested, the formula

$$1 + \frac{A}{C} = \frac{11}{\log\frac{72}{F}}$$

or

$$\log F = \log 72 - \frac{11}{1 + \frac{A}{C}}$$

gives us the value of the fluidity at the desired concentration.

A problem may make the calculations clearer: A certain viscometer has a viscometer constant of 1970 and a volume of 19.8 cubic centimeters. A sample of cotton weighing 0.0985 gram and containing 3.85% regain was dissolved and tested. The time of flow was 270 seconds. What is the fluidity of the sample in 0.5% solution?

$$\text{Dry weight of sample} = \frac{0.0985}{1.0385} = 0.0949 \text{ gram}$$

$$F = \frac{K}{0.94t} = \frac{1970}{0.94} \times 270 = 7.76$$

$$C = \frac{100W}{V - 0.7} = \frac{100 \times .049}{19.8 - 0.7} = 0.497$$

$$A = C\left(\frac{11}{\log\frac{72}{F}} - 1\right) = 0.497\left(\frac{11}{\log\frac{72}{7.76}} - 1\right) = 5.15$$

Then

$$\log F = \log 72 - \frac{11}{1 + \frac{A}{C}} = 1.857 - \frac{11}{1 + \frac{5.15}{0.5}} = 0.883$$

or

$$F = 7.6 = \text{fluidity in } 0.5\% \text{ solution}$$

Pure cotton cellulose in 0.5% solution will range in fluidity from 1–5, averaging about 2; it should never exceed 10 during manufacture. Mechanically disintegrated cotton will be about 40 with the end of service at about 35. Rayons in 2% solution will be about 7.5, and rayons damaged to the point of mechanical disintegration will be about 35. Rayon in 0.5% solution will be 36–40, and disintegrated rayons about 60. Silk and wool show a fluidity of about 65 in 0.5% solution, in case it is necessary to test mixed fibers. Typical values of the fluidity of rayons and cottons are shown in Table XXV.

The advantages of the fluidity test over the copper number determination are:

1. It shows incipient damage.
2. It shows damage even after an alkali boil or laundering.
3. There is a direct relationship between increase in fluidity and loss in strength. An increase in fluidity of 10 reciprocal poises corresponding to 20–25% loss in strength.

The disadvantages are:

1. A constant-temperature bath is necessary.
2. Cumbersome apparatus and a source of nitrogen gas are necessary for storing the cuprammonia solution in an inert atmosphere.
3. A long period of time is necessary to dissolve the samples.

In view of these advantages and disadvantages, the test is not much used unless a large number of tests are to be made as a routine or in research work where a very precise method of damage estimation is needed.

The use of organic bases such as dimethyldibenzylammonium hydroxide (*Triton F*) and cupriethylene diamine as solvents for the cellulose have been suggested and are apparently successful. These bases may be obtained already prepared and thus eliminate the tedious preparation of the cuprammonia solution. They still require precautions against oxygen damage during solution but are not as sensitive as cuprammonia.

Other uses for viscosity or fluidity determinations in the textile industry include:

1. Analysis of cotton-rayon mixtures. Assuming the fluidity of cotton in 0.5% solution to be 2 and viscose to be 36, the determination of the fluidity of a mixture gives a measure of the relative amounts of two constituents.
2. Liability to damage. Clibbens and Little have shown that a sample of cotton goods which has been heated in an oven at 110° C. for 18 hours and which has a rise in fluidity of less than 3 reciprocal poises contains no injurious chemicals; a rise of more than 3 reciprocal poises indicates chemicals which may cause damage on storing.
3. Degree of ripening of viscose. The viscose solution before spinning may be tested for *ripeness* by a viscosity determination. The falling ball method is generally used.
4. Degree of esterification of cellulose. The degree of nitration or acetylation may be determined by measuring the viscosity of the ester in an appropriate solvent.
5. Suitability of starch pastes for printing. It has been shown that a Stormer viscometer may be used for predicting the suitability of a starch paste for printing.
6. Damage to silk may be measured by viscosity (see Chapter 17), and more recently viscosity measurements have been used to determine damage to nylon.

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Chapter 16

DAMAGE TO WOOL

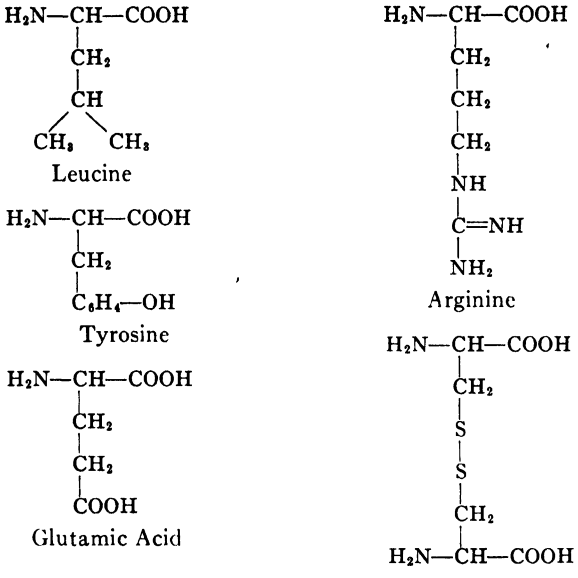
Introduction

In the case of wool, we have a fiber which is much more complex than cellulose and about whose structure and chemical reactions we know much less than we do in the case of cellulose. A complete hydrolysis of wool shows that it is a protein composed of a large number of amino acids of which the principal ones are given in Fig. 55. These and other amino acids are combined in long chains by peptide linkages, and these chains are thought to be folded in some such manner as shown in a, Fig. 56. These folded chains are further thought to be connected by side chains with a salt linkage (b, Fig. 56) and with cystine linkage (c, Fig. 56); of these side chains, the cystine linkage seems to be the most important. In cases of chemical attack on the wool, the points affected seem to be the peptide linkage, the disulfide linkage, and the amino groups; the disulfide linkage, is apparently the most sensitive to chemical attack.

The types of chemical treatment to which wool is exposed and which may cause damage are:

1. Acids (carbonizing, dyeing)
2. Alkalis (scouring, fulling, laundering)
3. Hot water or steam (crabbing, drying)
4. Reducing agents (reduction bleach, stripping)
5. Oxidizing agents (peroxide bleach)
6. Chlorination or bromination (non-shrinkage processes)
7. Light (on the sheep, in use)
8. Dry heat (drying, pressing)

The damage may manifest itself as uneven dyeing, tendering, harshness, or as an abnormal susceptibility to some process



Leucine

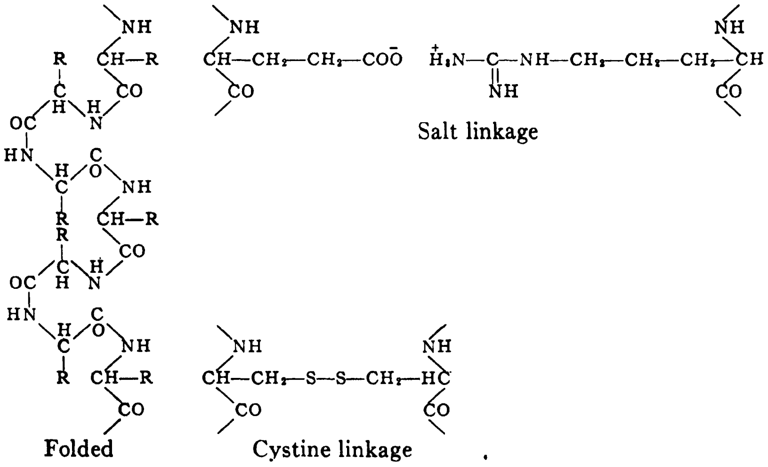
Tyrosine

Glutamic Acid

Arginine

Cystine

FIG. 55. Principal Amino Acids in Wool.



Folded peptide chain

Salt linkage

Cystine linkage

FIG. 56. Structure of Wool

which normally would not affect the wool seriously, such as finishing operations or laundering. The problem is complicated by the fact that practically all wools are damaged somewhat by light and alkali when they are sheared from the sheep, by the fact that the damage very often does not show up until after dyeing, so that color tests cannot be used, and by the fact that damage may occur in a process with practically no change in strength or handle until some other process is given the wool, for example, alkali damage may occur which does not show up until after an acid treatment (as in dyeing) or oxidation damage may occur but not show up until after an alkaline treatment (as in scouring or laundering).

The tests which have been used in determining the presence and the amount of damage are as follows:

1. Tests for physical properties
    a. Strength
    b. Stretch

These tests have already been discussed. They require large areas of damage and also require an undamaged sample for reference.

2. Microscopical examination
    a. Appearance
    b. Action with swelling agents

These examinations are probably the best methods of testing. They will be considered in the section on microscopical testing.

3. Dyeing or staining tests
    a. Benzopurpurin 10B
    b. Indigo carmine
    c. Kiton red G

These are not, of course, suitable for use on dyed samples.

    d. Methylene blue

This test may be used on dyed samples and may be made quantitative. The test is complicated and requires special apparatus.

4. Chemical color reactions

   a. Stannous chloride test

The author has had no success in using this test.

   b. Lead acetate
   c. Thiocyanate test

These are tests for bleached wool and are useful in such cases; they are, of course, not applicable to dyed wool.

   d. Pauly test

This is the best of the color tests and may be made quantitative if desired; it is not applicable to dyed wool.

5. Chemical-content tests

   a. Sulfur content
   b. Total nitrogen content

These tests require very precise analytical work since only a small difference is shown. An undamaged sample of the same wool is also required as a control.

   c. Amino nitrogen

This requires elaborate, special apparatus, and the results are not very reliable.

   d. Ammonia nitrogen

This is a quite satisfactory determination for certain types of damage which have not had any alkaline treatments subsequent to damage.

6. Solubility tests

   a. Soluble nitrogen

This requires a long time to obtain the results and two Kjeldahl determinations are necessary.

b. Solubility in dilute alkali

This is probably the simplest and best chemical test where the sample has not had an alkaline treatment after damage.

In view of the above advantages and disadvantages, we shall consider only the following in detail:

1. Qualitative tests
   a. Lead acetate
   b. Thiocyanate
   c. Pauly (may also be quantitative)
2. Quantitative tests
   a. Methylene blue
   b. Sulfur content
   c. Total nitrogen content
   d. Soluble nitrogen
   e. Ammonia nitrogen
   f. Solubility in dilute alkali

Qualitative Tests

LEAD ACETATE TEST

The lead acetate test of Smith and Harris is used as a test for bleached wool. On oxidation with peroxides, it is believed that the disulfide is oxidized to a series of compounds as follows:

(1) $R - S - S - R$ (cystine)
(2) $R - SO - S - R$
(3) $R - SO_2 - S - R$
(4) $R - SO - SO - R$
(5) $R - SO_2 - SO - R$
(6) $R - SO_2 - SO_2 - R$

Upon hydrolysis and treatment with lead acetate, compounds (1), (2), and (3) can form lead sulfide (black), whereas compounds (4), (5), and (6) cannot form sulfides and so remain white. The test is sensitive to pH, and the lead acetate solution must be buffered to pH 5 for best results.

The 0.5% lead acetate solution buffered to pH 5 is prepared as follows:

200 cubic centimeters of normal sodium acetate solution
20 cubic centimeters of 2.5% lead acetate solution
68 cubic centimeters of normal acetic acid
712 cubic centimeters of distilled water

The test is carried out by boiling approximately 1 gram of wool in 80 cubic centimeters of the lead acetate solution for 40 minutes. The sample is then rinsed and dried at room temperature. A black color indicates unoxidized wool, a white color indicates oxidized wool. This test is obviously not suitable for dyed wool.

In the case of light damage, oxidation occurs but not of the same type as in the case of peroxides; light-damaged wool, therefore, does not show up in this test, i.e., a dark color is obtained. The equations are believed to be:

$$\begin{aligned} R—CH_2—S—S—CH_2—R + HOH &= R—CH_2—SH \\ &\qquad + R—CH_2—SOH \\ R—CH_2—SOH &= H_2S + R—CHO \\ H_2S + 2O_2 &= H_2SO_4 \end{aligned}$$

since light damage has been shown to be due nearly entirely to a combination of hydrolysis and oxidation.

Alkali damage, which is believed to occur according to the first two of the above equations, also gives a black color and hence cannot be distinguished by the test.

THIOCYANATE TEST

This test for oxidation of wool shows oxidation by peroxides, chlorination or bromination, or by light. As it is a color reaction, it is not suitable for use on dyed samples. Three solutions are required:

Solution A: Equal parts by volume of acetone and distilled water, acidified by the addition of 1% by volume of 6N sulfuric acid.

Solution B: One gram of potassium thiocyanate in 30 cubic centimeters of solution A.

Solution C: One gram of ferrous ammonium sulfate and 15 cubic centimeters of 6N sulfuric acid in 50 cubic centimeters of solution A. Solution C must be prepared immediately before the test is carried out.

The test is carried out as follows: About 1 gram of wool is cut into small pieces and placed in a conical flask having a capacity of 125 cubic centimeters. In another flask is placed a similar sample of wool known to be unoxidized and in a third flask the reagents are used without any sample. In each flask are placed 40 cubic centimeters of solution A and 5 cubic centimeters of solution B. The solution is boiled for several minutes to displace air. The heating is discontinued; 5 cubic centimeters of solution C are added and the flask is stoppered. If a pink color develops in the flask containing no sample, distilled water is added until the color disappears; an equal volume of distilled water is then added to each of the other flasks. Unoxidized wool gives no color. Oxidized wool gives a pink color.

If a pink color develops before solution C is added, iron is present in the wool and should be removed by washing the wool in a number of changes of dilute sulfuric acid.

PAULY TEST

This is the most generally useful of the color tests since any kind of damage, whether mechanical or chemical, which will destroy, loosen, or remove the scales on wool will give the test. The theory of the test depends upon the fact that tyrosine, which is the only amino acid in wool which is also a phenol, is not found in the scales of the wool but only in the cortex. If we treat the wool, then, with diazotized sulfanilic acid, no color, or very little color, will be obtained except where the reagent can penetrate through the scales and allow the diazotized base to couple with the tyrosine and produce an orange color. The test may be made quantitative by dissolving the colored sample and comparing with a standard solution.

Solutions required:

Ten per cent sodium sulfanilate. This is made by dissolving 88.7 grams of sulfanilic acid in 1 liter of water containing 20.5 grams of sodium hydroxide.

Nine per cent sodium carbonate. This is made by dissolving 243 grams of crystalline sodium carbonate in water and making up to 1 liter.

Eight per cent sodium nitrite. This is made by dissolving 80 grams per liter of water.

Concentrated hydrochloric acid.

The reagent is prepared immediately before use as follows: Mix 10 cubic centimeters of 10% sodium sulfanilate with 5 cubic centimeters of 8% sodium nitrite. Then add 2 cubic centimeters of concentrated hydrochloric acid. Allow the reagent to stand for 1 minute before use.

The test is carried out by wetting a small sample of wool in 15 cubic centimeters of 9% sodium carbonate and then adding the prepared reagent to the mixture. After 10 minutes, the wool is removed and rinsed thoroughly. An orange-red color indicates damage.

To make the test quantitative, a sample weighing 0.1 gram is used and the test is carried out as above. The colored wool is then placed in a test tube, 4 cubic centimeters of 10% sodium hydroxide are added, and the test tube is heated in a water bath for 5 minutes. The dissolved wool is then transferred to a volumetric flask and made up to 25 cubic centimeters. The colored solution is then compared either visually or by means of a colorimeter with a 0.1% solution of Naphthalene Leather Brown BS which is arbitrarily called *100 units* of damage. Edwards has suggested that a better comparison color is obtained by the addition of 292.5 cubic centimeter of 0.1%

Polar Brilliant Red to a liter of 0.1% Naphthalene Leather Brown BS solution.

For most accurate results, a control of wool which is undamaged should be run along with the unknown sample since a certain amount of coloring is obtained on practically any sample.

Quantitative Tests

METHYLENE BLUE TEST

This test determines the amount of methylene blue absorbed at pH 7 by a sample of wool according to a procedure similar to that already described for damaged cellulose (Chapter 15). The methylene blue at pH 7 is the same as that already given and the wool is first given a standard acid wash. The wool is then divided, in a mill, into a powdery material which will pass a one-millimeter sieve, allowed to condition overnight and samples weighing 0.1 gram are weighed out. Another sample for regain determination is weighed out.

Portions of exactly 15 cubic centimeters of methylene blue solution are measured into centrifuge tubes having capacities of 40 cubic centimeters. The wool samples are added and allowed to stand for 24 hours. The tubes are then centrifuged. Ten cubic centimeters are pipetted out and titrated against naphthol yellow S. The methylene blue absorption in millimoles per 100 grams of dry wool is calculated, as in the case of cellulose. Undamaged wools have an absorption value of about 10, wool damaged by laundering (alkali) has a higher value, 16–18, and reworked wool fabrics (acid damage) a value of around 40. The method is said to be applicable to dyed samples.

The above test is not very critical for samples which are only slightly damaged but would be very useful to distinguish reworked wool if it were not for the elaborate apparatus and long periods of time necessary for testing. These latter disadvantages make the test useless for commercial purposes.

The increased absorption of methylene blue by acid damaged wool is believed to be due to a new and active acid group added by the wool on carbonizing:

In dilute sulfuric acid, $R—NH_2 + H_2SO_4 = R—NH_3—HSO_4$
On concentration by heating, $R—NH_3—HSO_4 = H_2O + R—NH—HSO_3$.

SULFUR CONTENT

The sulfur content of the wool has long been used in research work as a measure of the amount of damage to the wool, since any form of chemical damage tends to decrease the amount of sulfur in the sample. This is possible in research work where a sample of the wool before damage is available for comparison, but on an unknown sample the method is not useful since we do not know how much sulfur was originally present, and the amount of sulfur in the undamaged wool may vary from 3.1 to 4.0%.

Three methods of analysis are available for determining the sulfur content: Carius method, oxygen-bomb method, and the Benedict-Denis method. The Carius is perhaps the most accurate, the oxygen bomb is the simplest if the apparatus is available, the Benedict-Denis method requires no special apparatus or precautions. Since, in any case, a comparison with an undamaged sample must be made, if the result is to have any significance, it makes little difference which method is used. The Benedict-Denis method will be described.

Reagent: Dissolve 25 grams of crystalline copper nitrate, 25 grams of sodium chloride, and 10 grams of ammonium nitrate in 100 cubic centimeters of water.

About 0.2 gram of wool is warmed with a little pure sodium hydroxide solution until it just dissolves. A few drops of bromine are added, and after a few minutes, the solution is neutralized with nitric acid. Ten cubic centimeters of the reagent are then added, and the mixture is evaporated to dryness. The residue is heated to dull redness for 10 minutes, cooled, dissolved in dilute hydrochloric acid, and filtered. The sulfate in the filtrate is then determined by the usual quantitative procedure with barium chloride.

TOTAL NITROGEN CONTENT

This determination, like the sulfur content, is useful if a sample of the wool before treatment is available, since only a slight change in nitrogen content may occur and there is a large variation (16.16 to 17.07%) in the nitrogen content of various wools. Table XXVI shows the changes which may be expected in wool from different types of damage.

The total nitrogen content is determined by one or another of the various Kjeldahl determinations. A variation which has given satisfactory results is as follows: Regain is determined on a separate sample. Another sample of 0.7 to 0.8 grams is used for the digestion. The sample is placed in a Kjeldahl digestion flask with 20 cubic centimeters of concentrated sulfuric acid, 10 grams of powdered anhydrous potassium sulfate, 0.2 gram of copper sulfate, 4 grams of powdered pumice, and a little broken glass. The mixture is digested for 1 hour or until a clear melt is obtained; it is then dissolved in distilled water and cooled. An excess of sodium hydroxide solution is then added carefully. The flask is immediately connected with a Kjeldahl distillation apparatus, and the distillation is carried out for 30 minutes, the distillate being collected in 25 cubic centimeters of N/2 sulfuric acid. The excess sulfuric acid is then titrated with standard N/10 sodium hydroxide, using methyl red indicator.

SOLUBLE NITROGEN

This determination is not used to any great extent in this country, having been developed in Europe. The determination seems to be quite definite for alkali damage but involves two Kjeldahl determinations and a three-day treatment of the sample.

Two samples are taken. On one a Kjeldahl determination

TABLE XXVI
RESULTS OF TESTS FOR DAMAGE TO WOOL

	Normal Wool	*Acid Damage*	*Alkali Damage*	*Oxidation Damage*	*Chlorination Damage*	*Light Damage*
Lead acetate test	Black		Black	Light		Black
Thiocyanate test	No color			Pink	Pink	Pink
Pauly test	Little orange color	Orange	Orange		Orange	Orange
Methylene blue absorption, millimoles/100 gm	Low 10–11	Very high ca. 40	High ca. 18			
Ammonia nitrogen, mgms./gm	Low 0.15–0.20	High above 0.20		High		High
Solubility in dilute NaOH, %	Low 12–13			High over 18	High over 18	
Sulfur content, %	3.1–4.0 Av. 3.3		Decrease	Little change—decrease after alkali	Decrease	Decrease
Total nitrogen content, %	16.16–17.07 Av. 16.72	Decrease	Increase	Slight decrease	Little change	Decrease
Soluble nitrogen, %	Low 4–5		High ca. 32			

for total nitrogen is run. The other sample (0.1 gram) is treated for 3 days with a mixture of 8 cubic centimeters of water, 10 cubic centimeters of 1% hydrogen peroxide, and 2 cubic centimeters of N/2 potassium hydroxide. At the end of 3 days. the sample is filtered and washed, the combined filtrate and washings are made acid, and a Kjeldahl determination is run on the acid solution. The results are expressed as per cent of the nitrogen in the sample which dissolves on the above treatment. For undamaged wool, this is 4 to 5%, whereas for damaged wool it may be 32% or more.

AMMONIA NITROGEN

This determination is quite significant on samples which have not been exposed to alkali treatments after the damage occurred. It is especially valuable for identifying carbonized wool. The test is not convenient for use on a large number of samples at a time.

A five-gram sample of the wool is placed in a liter distilling flask with 300 cubic centimeters of water and 5 grams of magnesium oxide (free from carbonates). The flask is heated, and 150 cubic centimeters of liquid are distilled over into 25 cubic centimeters of standardized N/100 sulfuric acid. The excess sulfuric acid is then titrated with standardized N/100 sodium hydroxide, using methyl red indicator. The results are calculated to milligrams of ammonia nitrogen per gram of dry wool. Normal wool will give a value of 0.15 to 0.20, carbonized wool 0.20 to 0.70, and damaged wool may be even higher.

SOLUBILITY IN DILUTE ALKALI

This is perhaps the most generally useful test for certain types of damage (see Table XXVI). The test is affected to a certain extent by the form of the sample and the figures are given for loose wool; samples of yarn or cloth should be unraveled or picked apart. The test is simple to carry out, especially if a constant-temperature bath is available.

The wool is dried and weighed and is then treated with N/10 sodium hydroxide at 65° C. for 1 hour, the ratio of wool to solution being 1:100. The sample is filtered on a Buchner funnel or Gooch crucible, washed with about 2 liters of water, dried at 105° C., and weighed. The per cent loss in weight on the dry weight of the wool is calculated. This loss in weight will be normally 12–13%; above 18% shows definite damage, while between 13 and 18% is indefinite. The test, of course, is not suitable for use on wool which has been given an alkaline treatment after being damaged, or on wool which has been damaged by alkali.

According to Ryberg, this test may be simplified by using N/20 sodium carbonate and boiling for 30 minutes. The latter test is quicker and avoids the use of a constant-temperature bath. Normal wool will lose 10–11% in weight. Damage begins when the loss in weight exceeds 12%.

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Chapter 17

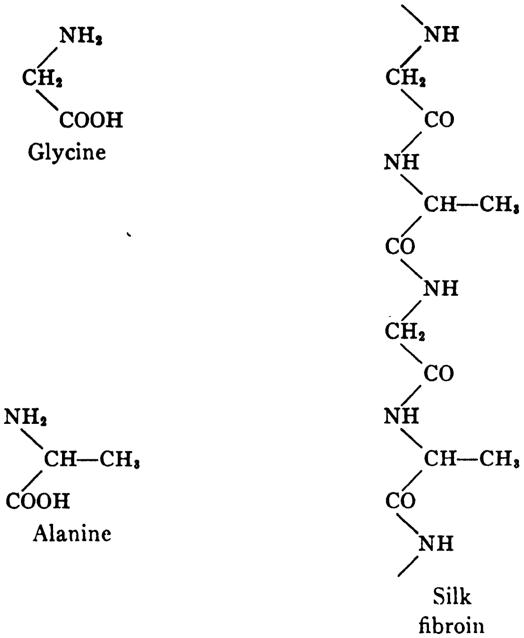
DAMAGE TO SILK

Introduction

The structure of completely degummed silk (fibroin), although not completely elucidated, is still much better understood than that of wool and is apparently much less complicated. Upon complete hydrolysis, the silk fibroin yields almost exclusively three amino acids. They are glycine (a, Fig. 57), alanine (b, Fig. 57), and tyrosine (Fig. 55). Of these three, glycine and alanine predominate. It will be seen from the formulae of these amino acids that the side chains contain no active groups and consequently, the salt and cystine linkages found in wool are lacking in silk. The fibroin, then, is considered as long chains of alternating glycine and alanine molecules held together lengthwise by peptide linkages and sideways by partial valence forces (a, Fig. 57). Therefore, silk is rather easily swollen and dissolved, but the peptide chains themselves are susceptible only to hydrolysis. Tussah silk is more resistant than cultivated silk to acids and alkalis but less resistant to steam.

The forms of damage which may occur to silk in processing are:

1. Acid damage. This consists of hydrolysis followed by decomposition.
2. Alkali damage. This consists of decomposition into a soluble form followed by hydrolysis.
3. Salt damage. This may be acid or alkaline damage according to the hydrolysis of the salt.
4. Oxidation damage
5. Light damage. This is apparently an oxidation.



Glycine

Alanine

Silk fibroin

FIG. 57. Structure of Silk Fibroin

6. Steam damage. This is hydrolysis, but not like acid damage.
7. Dry-heat damage

Tests which have been used to detect and estimate damage on silk are the following:

1. Physical tests such as strength and stretch tests. Wet strength seems particularly sensitive to damage.
2. Microscopic tests
3. Color reactions
    a. Zimmermann test
4. Chemical content
    a. Total nitrogen content
    b. Amino nitrogen content
    c. Ammonia nitrogen
5. Viscosity

The amino nitrogen is difficult to determine and requires elaborate apparatus. It will not be discussed further. However, the other tests will be considered in detail.

Tests

ZIMMERMANN TEST

Zimmermann's reagent consists of a solution of o-phthalaldehyde made acid with hydrochloric acid. This reagent gives no color with degummed and undamaged silk but gives a violet color with damaged silk or with sericin (silk gum).

TOTAL NITROGEN CONTENT

This determination is carried out by the Kjeldahl method in the same way as for wool (Chapter 16) and has the same disadvantages as in the case of wool. Normal silk contains 18 to 19% nitrogen (average 18.4%) and tussah contains somewhat less.

AMMONIA NITROGEN

This determination is carried out in the same way as it was described for wool (Chapter 16). Normal silk will give a value of 0.07 to 0.12 milligrams per gram of silk. Damaged silk will give a value greater than 0.2 milligrams per gram of silk.

VISCOSITY

This is the most satisfactory test for damage to silk, since it determines whether damage has occurred and gives some idea as to the amount of damage. The original method of Trotman and Bell has been modified by Tweedie. Any pipet-type viscometer may be used, including the one described for cuprammonia viscosity of cellulose (Chapter 15).

A solution of zinc chloride having a specific gravity of 1.67 at 20° C. should be prepared; this solution has a viscosity at 20° C. of 9.5 centipoises. Using this solution as the solvent, a 2.5% (weight/volume) solution of silk is made up, using the weight of silk at standard regain, that is, 2.50 grams of silk at standard regain are placed in a flask and 100 cubic centimeters of zinc chloride solution are added. The flask is stoppered and is placed in an oven at 45° C. for 3 hours to dissolve the silk. The flask is then cooled in water to 20° C. and the contents are placed in the viscometer. The time of flow at 20° C. is noted for the silk solution and for the zinc chloride without the silk. Since the addition of the silk does not materially alter the density of the solvent, the viscosity of the silk solution is equal to:

$$V = 9.5\frac{T}{t}$$

where

V = viscosity of the silk solution in centipoises
T = time of flow of the silk solution at 20° C.
t = time of flow of the zinc chloride solution with no silk dissolved, at 20° C.

Mechanical damage does not affect the viscosity of the silk but chemical damage will decrease it. The test may be carried out on dyed material and may also be carried out on weighted silk if the weighting is first removed by the procedure given in Chapter 12.

Undamaged silk will give viscosities of 19 to 24 centipoises; damaged silk will give values less than 19 centipoises, complete disintegration of the silk corresponds to 10–12 centipoises.

In carrying out this test, it is essential that the specific gravity of the zinc chloride solution be 1.67; a solution with a specific gravity of 1.62 will not dissolve the silk.

A later method for determining silk damage by fluidity is the method of Garrett and Howitt. In this method, a solution of 8% ethylenediamine and 6% cupric hydroxide is used to prepare a 10% solution of silk (weight/volume) which is then neutralized with an equal volume of 1.25N acetic acid. The fluidity may be determined with the Clibbens and Geake viscometer or with a U-type viscometer. Undamaged silk has a fluidity of 4; good-quality raw silks carefully degummed and washed have a fluidity of 5 to 8, a 10% loss in strength from any cause corresponds to about 20 reciprocal poises, and complete disintegration is shown at about 40.

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PART III

MICROSCOPICAL TESTING

Chapter 18

IDENTIFICATIONS

Introduction

Since it would be impracticable here to discuss fully the construction and use of the microscope and accessories, it will be assumed that the reader is acquainted with the microscope and has sufficient practice on methods so that detailed descriptions of operations need not be given. Before any microscopical testing is done, it is essential that a knowledge of fundamentals be obtained from the references given here and that sufficient practice be obtained on samples of known composition and properties so that the technique of the art is familiar. No photomicrographs are given for the recognition of the fibers because there is so much variation in individual fibers that a false idea of what to look for might be obtained. Instead, it is strongly recommended that a collection of known samples be made and kept available for comparison, so that not only the ordinary appearance but any normal variations in appearance may be known.

We have seen in Chapter 14 that it is impossible to recognize with certainty every fiber by chemical means alone. The microscope alone will do this, but a combination of chemical and microscopical methods usually removes all doubt. An additional advantage of microscopical testing is the fact that very small samples may be used and that the samples are not destroyed.

Identification of Starches

The identification of a starch depends upon three characteristics: the size, the shape, and the presence or absence, as well as the shape, of a marking on the starch grain called the hilum. Table XXVII gives the appearance of the more common types of starch when freshly prepared. It will be noted that there are three sizes of starch grains (small, medium, and large), three shapes (polygon, circular, and oval) of which the circular and oval may be truncated, i.e., cut off, and three types of hilum (dot, transverse, i.e., one line, and stellate, i.e., two or more intersecting lines).

TABLE XXVII
MICROSCOPICAL APPEARANCE OF STARCHES

Type of Starch	*Size*	*Shape*	*Hilum**
Rice	Small 3–10 μ	Polygon	None
Corn	Small, medium 10–30 μ	Polygon	Stellate
Wheat	Small, large 1–8 μ, 15–45 μ	Circular	None
Tapioca	Small, medium 5–26 μ	Circular (truncated)	Dot
Potato	All sizes	Circular (S, M) Oval (L)	Dot on large grains
Arrowroot	Medium, large 35–70 μ	Oval	Dot or transverse
Sago	Medium, large 30–65 μ	Oval (truncated)	Dot or transverse

* The descriptions in this column refer only to starch which has not been altered by age, heating, or chemical treatment.

The identification is fairly easy on a freshly prepared starch but is complicated on an old starch, a boiled starch, or a chemically modified starch (dextrin) by the fact that, on these treatments, the hila change from dot to transverse, transverse to stellate, and stellate to ruptured grains. Too much reliance, therefore, must not be placed on the appearance of the hilum unless the history of the starch is known. A magnification of 100× should be used for most work, with a 400× magnification available for closer examination of individual grains. A comparison of the unknown with known samples is always desirable. In the case of greatly decomposed starches, identification must depend upon the examination of the few grains that are left intact.

Identification of Fibers

In Chapter 14, in considering the qualitative tests for the various fibers, it was strongly recommended that the microscope be used to amplify and check the chemical tests and also to identify members of groups which could not be differentiated by chemical means.

CLASSIFICATION

In considering the microscopical analysis of fibers, we shall classify the fibers according to origin, rather than according to chemical constitution, into:

1. Hairs
   a. Sheep family (wool)
   b. Goat family (mohair, cashmere, common goat hair)
   c. Camel family (camel, llama, alpaca, vicuna hair)
   d. Rabbit family (rabbit hair)
2. Silks (true silk, tussah silk)
3. Vegetable-seed hairs (cotton, mercerized cotton, kapok)
4. Bast (stem) fibers (flax, ramie, hemp, jute)
5. Artificial fibers
   a. Cellulose base (rayons)—acetate, saponified acetate, cupra, viscose, high-tenacity viscose
   b. Protein base (prolons or azlons)—casein wool, soybean fiber
   c. Synthetic (synthons)—vinyl fiber, nylon
   d. Silicates (glass)

EXAMINATION

Hairs are best examined in a mount of glycerin or colorless mineral oil and at a magnification of 100×. Higher magnification may be required for individual fibers. The mounting technique of Manby may be used. The method of Hardy for making casts of the fibers is useful on dyed or heavily pigmented hairs to study the scale structure.

In examining the hairs for identification, the following features should be looked for:

1. Scales
   - a. Shape of outline. Llama, alpaca, and vicuna are indistinct. Camel hair has a diagonal edge. The other hairs are irregular.
   - b. Amount of projection. Wool, cashmere, and rabbit hair have a marked projection of the scale from the fiber outline. Other hairs are smoother in outline.
2. Medulla
   - a. Presence. All beard hairs have a medulla, and some wool hairs have it also. Llama, alpaca, and vicuna wool hairs have a medulla as well as the beard hairs.
   - b. Size. Cashmere and camel hair medullas are larger than other hairs.
   - c. Form. Most hairs have a continuous medulla. Many camel wool hairs have an interrupted form. Llama beard hairs have a double medulla. Rabbit hair medullas are in the form of a chain of air pockets.

3. Color granules. The complete absence of color granules in all individual hairs is strong evidence of wool or mohair. Other hairs may have some fibers with no color, but some of them always contain color granules.

4. Diameter of fibers. All hairs have a large range of fiber diameters but common goat hair is composed of nearly all coarse hairs. The wool hairs of camel, cashmere, and vicuna are especially fine. See Table XXXII.

Table XXVII gives a comparison of the microscopical properties of the textile hairs, but this table should also be supplemented by authentic samples for comparison with unknowns. Of all of these, wool and mohair are by far the most common; and the hardest distinction to make is between certain wools and mohairs. Skinkle's scale length method may be used for this: if the visible scale length is below 17 μ, the fiber is wool; if above 18.5 μ, it is mohair; if between 17 and 18 μ, it is probably wool and between 18 and 18.5 μ, it is probably mohair. In this last

TABLE XXVIII
MICROSCOPICAL APPEARANCE OF HAIRS

Name and Natural Color	*Wool Hairs*	*Beard Hairs*
Wool (white)	Scales project, loosely arranged D = 12–70 μ S = less than 18 μ S^3/D = less than 140	Not many, except in *luster wools*
Mohair (white)	Scales close to surface D = 14–90 μ S = greater than 18 μ S^3/D = greater than 160	Not many
Cashmere (white, grey, brown)	Scales project slightly, serrated. White and gray have striations; brown has color granules. D = 14–16 μ (average) S = 14–17 μ	Medulla makes up large part of fiber D = 30–150 μ
Common goat hair (white, grey)	D = 7–20 μ	Mostly beard hairs Roots present D = 50–200 μ
Camel hair (brown)	Scales diagonal Striated, color granules Some interrupted medullas D = 9–40 μ, 18–24 μ (average) S = 11–16 μ	Wide, continuous medullas D = 30–120 μ
Llama (white, brown)	Scales indistinct Striated, color granules Few non-medullated fibers D = 10–60 μ, 20–27 μ (average)	Double channel medullas
Alpaca (white, grey, black, brown)	Scales indistinct Striated, color granules Indistinct hollow medulla Few non-medullated fibers D = 10–60 μ, 26–27 μ (average)	
Vicuna (white, fawn, brown)	Scales indistinct Striated, color granules Few non-medullated fibers D = 6–25 μ, 12–14 μ (average)	D = 68–80 μ
Rabbit (white, gray, brown)	Scales project Medulla contains continuous chain of air pockets. D = 13–15 μ (average)	

D = diameter of fiber in microns.
S = visible scale length in microns.

case, measure also the diameter and calculate S^3/D; if this last value is below 140 it is wool and if above 160 it is mohair.

Lamb's wool and hogg wool may be identified by the presence of natural tapering fiber ends. Pulled wool shows occasional root ends, some broken ends, and occasionally crystalline particles which adhere to the fiber. Shoddy or reworked wool may be recognized by the torn or broken ends, cracked and broken bends, and usually by the large number of colors present.

The silks are easily distinguished from each other. True silk is a narrow fiber with no markings; tussah silk is a broad fiber with longitudinal striations, particles of colored pigment, and frequently with parallel pairs of diagonal lines. The cross-sections of true silk are quarter circles or half circles (with sharp corners rounded off); the cross-sections of tussah are very much flattened circles.

In the vegetable-seed hairs, cotton is distinguished easily from kapok by the fact that the cotton is a broad flat fiber with frequent twists. A broad lumen is present in the cotton but the walls of the fiber are quite thick. Kapok consists of a circular hollow tube with a broad lumen, very thin walls, and frequent bends but no twist. Mercerized cotton is rounder, without twist, and the lumen has disappeared or become very narrow. Incompletely mercerized cotton has fibers of both types and should be given the *deconvolution count* (see Chapter 19) for recognition.

Table XXIX gives the characteristics of the vegetable-stem fibers or bast fibers. These fibers are found in bundles, cemented together, and it is necessary to break up the bundles to obtain the individual fibers before recognition may be made. It will be noted that jute is distinguished from the others by the absence of joints, and flax is distinguished by the pointed end. If chemical tests can be used, the longitudinal appearance is all that is necessary, but if identification must be made by the microscope, then cross-sections should be prepared. The use of polarized light aids in deciding quickly whether or not joints are present. Hardy's sectioning device is useful for preparing cross-sections. It will be noted that flax line and tow may be distinguished by the width of the lumen. Bast fibers are best mounted in water and examined at about 200× magnification.

TABLE XXIX
MICROSCOPICAL APPEARANCE OF BAST FIBERS

	Joints	*Lumen*	*Natural Ends*	*Shape of Cross-section*	*Cross-section Lumen*
Flax line	Present	Narrow	Pointed	Sharp polygons	Small, round
Flax tow	Present	Broad	Pointed	Sharp polygons	Larger, oval or round
Ramie	Present	Broad	Blunt	Elongated rounded polygons	Polygons with radial cracks
Hemp	Present	Broad, indistinct	Blunt	Rounded polygons	Cleft-shaped
Jute	Absent	Broad, variable	Blunt	Sharp polygons	Oval or round

Table XXX gives the microscopical appearances of the artificial fibers. Both longitudinal and cross-sectional mounts must be made, and it is almost a necessity that chemical tests be used to amplify the microscopical appearances. The appearances of the newer fibers are given as they are now made, but it is possible that changes in manufacture may alter these appearances; therefore some known samples are necessary for comparison.

In first looking at a fiber, it is generally possible to assign it to one of the several groups as shown in Table XXXI. After that, the observation of further details will generally identify the fiber, but it is better and safer to check results by preparing a cross-section and making suitable chemical tests as given in Chapter 14.

TABLE XXX
MICROSCOPICAL APPEARANCE OF ARTIFICIAL FIBERS

Fiber	*Appearance of Longitudinal Mount*	*Appearance of Cross-section*
ACETATE	False lumen	Bulbous, indented
SAPONIFIED ACETATE	Same as acetate	Same as acetate
CUPRA	No markings	Small, nearly round
VISCOSE	Numerous striations	Diverse shapes, but always serrated
HIGH-TENACITY VISCOSE	Indistinct striations	Diverse shapes, indistinctly serrated
CASEIN	Smooth, some striations, some *pitting;* transitory diamond pattern when mounted in concentrated or 50% sulfuric acid	Nearly round, occasional air-filled small lumen
SOYBEAN	Smooth, some striations, some *pitting;* transitory diamond pattern when mounted in concentrated sulfuric acid but no diamond pattern (or very small) in 50% acid	Nearly round, occasional air-filled small lumen
VINYL	False lumen	Dumbbell shaped
NYLON	No markings	Small, round
GLASS	No markings	Small, round

Note: The above descriptions of appearance are for the lustrous types of fiber. The delustered fibers have the same shapes but contain numerous small particles of delustering agent.

TABLE XXXI
MICROSCOPICAL RECOGNITION OF FIBERS

Make Longitudinal Mount of Fiber in Mineral Oil and Examine at 100 × Magnification

Scales Present	*Twisted or Bent*	*Joints Present*	*Longitudinal Striations*	*No Markings*
HAIRS	COTTON KAPOK	FLAX RAMIE HEMP	TUSSAH JUTE ACETATE SAPONIFIED ACETATE VISCOSE HIGH-TENACITY VISCOSE CASEIN SOYBEAN VINYL	SILK MERCERIZED COTTON CUPRA NYLON GLASS
See Table XXVIII	Cotton: twisted, thick walls. Kapok: bent, thin walls.	See Table XXIX	Tussah: ribbon shape, color granules, diagonal marks. Jute: broad lumen, polygonal cross-section. Acetate: few striations, apparent lumen, nearly invisible in oil.	Mercerized cotton: remnants of lumen. Silk: triangular cross-sections. Cupra, Nylon, Glass: Round cross-sections. See Table XXX.

TABLE XXXI (*Continued*)

Scales Present	*Twisted or Bent*	*Joints Present*	*Longitudinal Striations*	*No Markings*
			Saponified acetate: same as acetate, visible in oil. Viscose; high-tenacity viscose: numerous striations, serrated cross-sections. Casein and soybean: indistinct striations, *pitting*, round cross-sections. Vinyl: apparent lumen, dumb-bell-shaped cross-section. See Table XXX.	
	Confirm kapok with chemical test.	Confirm hemp with chemical test.	Confirm with chemical tests (Table XXII). May also confirm by refractive indices.	Confirm with chemical tests (Table XXII). May also confirm by refractive indices.

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Chapter 19

QUANTITATIVE MICROSCOPICAL DETERMINATIONS

Wool Grading

The standard method of grading wool now consists in the determination of the average diameter of the hairs together with an estimation of the range of hair sizes. Two general procedures are available:

1. Preparation of a cross-section and determination of the diameter. This is too slow and complex and usually is not done.

2. Determination of the diameter from a longitudinal mount. The apparatus used may be:

a. An ordinary microscope fitted with a disc or filar micrometer, suitably calibrated.

b. The Bausch & Lomb projection apparatus and calibrated wedge.

c. The Zeiss Lanameter with scale. This is the European method.

MICROMETER METHOD

In the micrometer method, fibers are mounted on a slide in glycerin, and by means of a mechanical stage each fiber is brought into the field and its diameter measured with the micrometer. Usually 100 fibers are all that are necessary to give a good average, but if the sample is very variable, 200 or even 400 fibers may be necessary. The standard error and variation should be calculated; a variation of 20%, or more, usually indicates a blend. Table XXXII gives the range of the average diameters of each quality of wool and mohair, as well as the other common fibers. If the distribution of diameters is required as well as the average diameter, this method is rather slow,

TABLE XXXII
AVERAGE DIAMETERS OF FIBERS

Fiber	*Grade*	*Quality*	*Average Diameter Microns*
Wool	Fine	80's	18.1–19.5
Wool	Fine	70's	19.6–21.0
Wool	Fine	64's	21.1–22.5
Wool		62's	22.6–24.0
Wool	½ Blood	60's	24.1–25.5
Wool	½ Blood	58's	25.6–27.0
Wool	⅜ Blood	56's	27.1–29.0
Wool	¼ Blood	50's	29.1–31.5
Wool	¼ Blood	48's	31.6–33.2
Wool	Low ¼ Blood	46's	33.3–34.7
Wool	Common	44's	34.8–36.5
Wool	Braid	40's	36.6–38.7
Wool	Braid	36's	38.8–41.3
Mohair	Baby kid	40's	23.0–25.0
Mohair	Super kid	36's	25.0–27.0
Mohair	Kid	32's	27.0–30.0
Mohair	Super first	28's	30.0–34.0
Mohair	First	24's	34.0–40.0
Mohair	Second	20's	40.0–50.0
Mohair	Third	Low	50.0–60.0
Cashmere			15.0–16.0
Camel hair			17.0–23.0
Llama			20.0–27.0
Alpaca			26.0–28.0
Vicuna			13.0–14.0
Rabbit			13.0–15.0
Silk			10.0–13.0
Tussah			ca. 28.5
Cotton			16.0–21.0
Flax			15.0–17.0
Jute			15.0–20.0
Hemp			18.0–23.0
Kapok			21.0–30.0
Ramie			25.0–30.0

since 400 to 1600 fibers must be measured, and the wedge method is then used.

WEDGE METHOD

The wedge method is used primarily for the determination of the quality of wool top where the specifications call for a certain minimum number of fine fibers, a certain maximum number of coarse fibers, as well as the average diameter for each grade of wool (Table XXXIII). This is the official standard method of the A.S.T.M. and the government, and it is now in use by all large concerns who buy or sell large amounts of wool. The apparatus consists of a projection microscope with a moveable stage and an objective and ocular which will give a magnification of 500× when projected the proper distance. The measuring device is a sheet of white cardboard with three wedges printed upon it covering the ranges 10–30 μ, 30–50 μ, and 50–70 μ. Samples 2 inches long are cut from the wool, the samples are opened out so that each fiber is freed from the others, mounted across a slide in glycerin, and covered with a cover-glass. The slide is placed on the stage of the microscope, the image projected on the wedge ruler, and the ruler turned until the image of the fiber to be measured is projected between the two lines of the wedge. The place where the wedge has exactly the same width as the image of the fiber is marked; 100 fibers are recorded on each wedge. Table XXXIII gives the number of measurements to be made; the measurements are then averaged and the percentage in each range calculated. Comparison with Table XXXIII then gives the quality.

Maturity of Cotton

Two methods are available for this determination:

1. Swelling method. This is the A.S.T.M. standard method. It requires no special apparatus.

2. Polarized-light method. This is the quicker, probably better method. It requires a polarizing microscope and selenite plate.

TABLE XXXIII
DIAMETER DISTRIBUTION REQUIREMENTS FOR WOOL TOP

Grade	Number of Fibers Tested	Average Diameter Microns	*Minimum Per Cent of Fine Fibers*				*Maximum Per Cent of Coarse Fibers*							
			10–20 μ	*10–25 μ*	*10–30 μ*	*10–40 μ*	*25.1–30 μ*	*25.1–40 μ*	*30.1–40 μ*	*30.1–50 μ*	*40.1–50 μ*	*50.1 and Over*	*40.1–70 μ*	*60.1–80 μ*
80's	400	18.1–19.5	60	92			8		0.25					
70's	400	19.6–21.0	50	84				16	2		0.25			
64's	600	21.1–22.5	36		94				6		0.33			
62's	600	22.6–24.0	27		88				12		0.50			
60's	800	24.1–25.5	18		83				17		0.50			
58's	800	25.6–27.0	16		74					26	2	0.75		
56's	1200	27.1–29.0	9		64					36	5	1.00		
50's	1200	29.1–31.5	4		45					55	10	1.25		
48's*	1600	31.6–33.2			34	77							23	2
46's*	1600	33.3–34.7			26	70							30	2
44's*	1600	34.8–36.5			22	63							37	3
40's*	1600	36.6–38.7			16	55							45	4
36's*	1600	38.8–41.3			12	44							56	4

* These standards are tentative.

SWELLING METHOD

In the swelling method, two groups of 100 fibers each are examined. The fibers are straightened, placed parallel on a slide, covered with a cover-glass, and flooded with 18% sodium hydroxide. The slides are then placed on the stage of a microscope equipped with a disc or filar micrometer and examined at the mid-portion of the fibers. As successive fibers come into view, they are counted, and the number of thin-walled immature fibers are also counted. Thin-walled fibers are defined as fibers, which on treatment with 18% sodium hydroxide, show a fiber wall thickness of less than half the width of the adjacent lumen at the widest part of the fiber ribbon, that is, the lumen is greater than one-half of the total width; all other fibers are classed as mature or thick-walled. Usually, it is plain whether the fiber is immature or mature. However, in doubtful cases, the micrometer is used. The percentage of immature fibers is calculated.

POLARIZED-LIGHT METHOD

In the polarized-light method, fifty fibers are taken for each test and are mounted substantially parallel with each other between two slides which are fastened with tape; no mounting medium is necessary. A polarizing microscope is used with a selenite plate placed between the crossed polarizer and analyzer (just above the polarizer, or above the ocular and below the analyzer) with the slow direction (indicated by the arrow or dot) at 45° to both the polarizer and the analyzer. The background will then be First Order Red or Red I. The slide is placed on the stage which is then rotated until the fibers are parallel to the slow vibration of the selenite plate. Each fiber which is clear purple or deep blue or a combination of both is given two demerits (immature); fibers which are blue or blue-green or both are given one demerit (partially immature); fibers which are yellow or yellow-green are given no demerits (mature). Since 50 fibers are examined, the total number of demerits is the percentage of immaturity. Several tests may be necessary.

Mercerization of Cotton

DECONVOLUTION COUNT

The simplest microscopical determination of the degree of mercerization is by the deconvolution count. In this test, fragments of cotton 0.2 millimeters long are cut, most conveniently by two razor blades set in a holder with the edges 0.2 millimeters apart. The fragments are teased apart, mounted in mineral oil, and covered with a cover-glass. A number of fields is then examined, and the total number of fibers, as well as the number of fibers which are free from twist or convolution, is counted. A fiber is free from convolution if there is less than 90° turn of the fiber. The percentage of deconvoluted fibers is then calculated. Unmercerized fibers are always below 16% and usually below 13%; values greater than 16% indicate mercerization in varying degrees. This test is useful in comparing different samples, in detecting mercerization in doubtful cases, and in detecting uneven mercerization in streaky dyeing.

Damage to Cotton

Two methods are available here:

1. Swelling test of Fleming and Thaysen
2. Congo red test of Bright

SWELLING TEST

In the swelling test, a small sample is shaken for 2 to 3 minutes with a mixture of equal volumes of carbon disulfide and a 15% (by weight) sodium hydroxide solution. Undamaged fibers show beadlike swellings; damaged fibers are swollen uniformly and do not show the beads. The number of damaged and undamaged fibers is counted, and the percentage of damaged fibers is calculated.

CONGO RED TEST

In the Congo red test, about 0.1 gram of cotton is placed in a filter flask with water and as much air as possible is withdrawn by a filter pump. The cotton is squeezed gently to remove most of the water, placed in 25 cubic centimeters of an 11% (by weight) sodium hydroxide solution, shaken, and allowed to stand 5 minutes. It is then washed rapidly in water, placed in a saturated (2%) solution of Congo red, and shaken at intervals for 6 minutes. The cotton is then removed, shaken with changes of water until there is no pink color in the water. The cotton is then placed in 18% (by weight) sodium hydroxide, teased out, and mounted with the same liquid. It is then covered with a cover-glass and examined at 100× magnification. In the case of mechanical damage the fiber is bruised or cut, the cellulose protrudes and is deeply stained. In the case of acid damage, the fiber has irregular red patches. In the case of heat or fungus damage, the fiber has red spiral bands.

Damage to Wool

In detecting damage to wool and, to a certain extent, identifying the type of damage two tests may be used;

1. Allworden's reaction using chlorine water or bromine water
2. Von Bergen's swelling test using N/10 sodium hydroxide

ALLWORDEN'S REACTION

In Allworden's reaction, the reagent used is saturated chlorine water or saturated bromine water; the bromine water is more convenient to prepare and keep. The fibers are placed on a slide and mounted in the saturated bromine water. If the wool is undamaged, globules will appear on the surface in 5 to 30 minutes. If the wool is damaged by alkali or light, swelling of the fiber may occur but globules will not appear.

VON BERGEN'S SWELLING TEST

In Von Bergen's method, the reagent used is N/10 sodium hydroxide. The fibers are mounted in water, and the diameters are measured with a disc or filar micrometer. Two drops of the reagent are placed at the rim of the cover-glass. The water is sucked out with filter paper applied at the opposite side. This procedure is repeated once more, and the diameter of the fibers is measured again in a few minutes. The swelling is expressed as per cent of the diameter in water. Normal wools swell 10 to 13%; wools damaged by oxidation, chlorine, light, or acid swell more than 15%; a swelling of greater than 20% corresponds to serious damage, and a swelling of 100% or more corresponds to complete destruction of valuable properties.

Analysis of Fiber Blends

In determining the quantitative composition of fiber blends, chemical methods should be used if the sample is large enough and the mixed fibers are chemically separable. If the conditions make a chemical analysis impossible, then the microscopical method may be used but only approximate results may be expected.

If we let

n_a = number of fibers of A
n_b = number of fibers of B
g_a = mean hair weight of A in milligrams $\times 10^{-5}$ per centimeter
g_b = mean hair weight of B in milligrams $\times 10^{-5}$ per centimeter

then

$$\text{Per cent of A} = \frac{100 n_a g_b}{n_a g_a + n_b g_b}$$

and, in order to obtain the composition of the mixture, we need to know the number of fibers of each kind as well as the average hair weight of each. An approximate relationship between the hair weight and the diameter (or width) of the fiber exists, as follows:

$$g = \frac{\pi}{4} S(KD)^2 = \frac{\pi}{4} SK^2D^2 = kD^2$$

where

g = hair weight in milligrams $\times 10^{-5}$ per centimeter
S = density of the fiber in grams per cubic centimeter

D = mean observed diameter (or width) in microns

K = a constant of form and is defined as $K = \frac{d}{D}$

where

d = true effective diameter of the fiber

For certain fibers (wool, mohair, cupra, nylon), K is equal to 1.0 or close enough; in the other fibers, K may have different values. The combination of K, S, and the value of $\pi/4$ is lumped together in another constant (k). Table XXXIV gives the value of S, K, and k for the common fibers and by means of this table the hair weights may be approximated.

TABLE XXXIV
CONSTANTS FOR CALCULATION OF HAIR WEIGHT FROM DIAMETER

Fiber	*S* *Density (Grams per Cubic Centimeter*	*K* *Constant of Form*	*k* *in the Equation* $g = kD^2$
Wool, mohair, etc.	1.32	1.0	1.04
Silk	1.25	0.85	0.71
Cotton	1.50	0.71	0.60
Cupra rayon	1.52	1.0	1.19
Linen	1.50	0.70	0.58
Viscose rayon	1.52	0.82	0.80
Acetate rayon	1.33	0.90	0.86
Casein	1.29	0.84	0.71
Nylon	1.14	1.0	0.89

The following case will show the procedure and calculations involved: A yarn composed of a mixture of viscose and wool is to be analyzed. The yarn is cut across with a razor blade, and a section about ¼ inch in length is cut out. The fibers are teased out on a slide; a drop of mineral oil and a cover-glass are added. This is done on several slides, and the results are combined. On examination, fifty-four wool fibers were found with an average diameter of 22.5 μ and forty-seven viscose fibers with an average diameter of 20.5 μ.

a. Wool:

$n_a = 54$

$D_a = 22.5\ \mu$

$g_a = 1.04D^2$ (Table XXXIV) $= 527$ milligrams $\times\ 10^{-5}$ per centimeter

$n_a g_a = 54 \times 527 = 28{,}400$

b. Viscose:

$n_b = 47$

$D_b = 20.5\ \mu$

$g_b = 0.80D^2$ (Table XXXIV) $= 336$ milligrams $= 10^{-5}$ per centimeter

$n_b g_b = 47 \times 336 = 15{,}800$

$$\text{Per cent wool} = \frac{100 \times 28{,}400}{28{,}400 + 15{,}800} = \frac{100 \times 28{,}400}{44{,}200} = 64$$

The method is least accurate where cotton is one of the components since the constant of form (K) may vary considerably from the recorded value. The method seems to be most accurate, as well as of most value, in blends of animal hairs such as wool and mohair or wool and camel hair. Here, the constants K, S, and k are the same and the formula may be written:

$$\text{Per cent of A} = \frac{100 n_a D_a{}^2}{n_a D_a{}^2 + n_b D_b{}^2}$$

and Table XXXIV is not necessary. The individual hairs may be identified as they are examined by use of the visible scale length as described in Chapter 18. This case is especially important since no method of chemical analysis is available.

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